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Adhesives for Bonding RSI Tile to GrPI Structure for Advanced Space Transportation Systems

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FOREWORD

This report was prepared by the Satellite Systems Division of Rockwell International under Contract NAS1-15152 for the Langley Research Center, National Aeronautics and Space Administration. The contract was initiated November 11, 1977. This report covers work from that date through December 20, 1978.

The program at Rockwell International was directed by the Design and Materials Group under the supervision of L.L. Bissing. The work was coordinated by K.E. Smith. Materials screening, mixture formulation, and exploratory testing were conducted at the Science Center under direction of Dr. C.L. Hamermesh, principal investigator, assisted by Mrs. Carolyn M. McArthur and J. Ratto. Verification testing was by the Laboratories and Test Group at Space Division under the supervision of Stanley Kritzer. The test program was conducted by P.A. Hogenson. Chemical characteristic tests were conducted by D.W. Houston, Jr..

Outline specifications were prepared by R.L. Long; these are included as Appendix C.

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ILLUSTRATIONS

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1. INTRODUCTION

The primary structure of the Shuttle orbiter is presently built of aluminum alloy having a design temperature limit of 350F (177C). A thermal protection system (TPS) consisting of fused silica tiles is bonded to the outer surface to protect the skin from temperatures that may reach 2650F (1454C) during reentry. As a key element of the Project CASTS (Composites for Advanced Space Transportation Systems), serious consideration is being given the use of filamentary composites as the structural material for selected orbiter components, such as the aft body flap. Graphite/polyimide (Gr/PI) laminates, which are being developed under the CASTS Program, have the capability of sustained operation at 600F (316C). This would provide the potential for weight savings, with a corresponding increase in payload capability. Weight savings would result primarily from the use of thinner tiles. The possibility also exists of eliminating the strain isolation pad (SIP) which is now bonded between the tiles and the orbiter skin. This would result in further weight savings and reduced manufacturing costs.

The limiting factor in taking full advantage of the 600F (316C) capability offered by the composite is the adhesive used in the TPS. The RTV 560 silicone adhesive now used has a maximum design temperature of 500F (260C). Prior to undertaking the current contract, the Satellite Systems Division, supported by Rockwell's Science Center, conducted an experimental program aimed specifically at developing adhesive systems to operate over a temperature range of -300F to +700F (-184C to 370C). The results of this effort are presented in Reference 1. Ability to perform over this range would provide adequate margin for the

foreseeable future. Emphasis was placed on developing polymer systems that could be bonded at room temperature by techniques compatible with manufacturing procedures to be applied during initial construction and in the field. This effort provided a starting point for initial screening of adhesive candidates for development under the current contract program.

2. SUMMARY

The main objective of the program was to develop a system for bonding RSI tiles to a Gr/PI composite substrate which would withstand the full range of environmental conditions imposed on the orbiter. While it was recognized that complete environmental testing to verify this capability was beyond the program scope, it was intended to provide high confidence, in the form of test data, that a bonding system with such capability had been developed. The program was successful in satisfying this objective. The bonding system, designated RA 59, consists of a mixture of glass (sesquisiloxane, SQS) resin in RTV 560 silicone. RA 59 has desirable processing characteristics similar to RTV 560 and, based on limited data, has the capability of withstanding all environmental and operating conditions required of the orbiter. It has material properties similar to the RTV over the normal operating range for RTV (-175F to +500F) but extends the upper operating range to 700F. Both adhesives appeared to perform satisfactorily down to -300F when used to bond RSI tiles directly to a Gr/PI composite substrate.

While results of most recent tests by both lap shear and flatwise tensile test methods exhibit considerable data scatter, a significant number of data points for the RA59 are in the 65-psi failure range both when tested at, and after exposure to, 700F. This is over two times the best shear and tensile values obtained with RTV 560 at this temperature. Perhaps the most important overall conclusion to be drawn is that with a thorough understanding of the critical parameters involved, the higher values should be obtained

consistently with the RA59. This would be of particular significance if higher strength tiles were to be used in a "hard-bonded" configuration as is now being considered for the aft body flap.

While not crucial to qualification, it would be highly desirable to develop an understanding of the chemical interractions involved in the mixture. Such understanding could lead to both optimization of materials and better consistency of the product. It is recommended therefore that the current program be continued to provide this information prior to undertaking a program of flight qualification.

This report describes the initial materials screening, formulation and exploratory testing conducted by the Science Center followed by the results of verification tests conducted by the Space Division. This effort complies with the Contract Statement-of-Work (Reference 2), Tasks I through IV. In addition, outline specifications for processing and procurement are provided in response to Tasks V and VI. Sufficient data on materials and processing could not be obtained to support preparation of detail specifications.

The original program plan included both direct bond and SIP (strain isolation pad) applications. By mutual agreement, SIP was dropped from consideration during the early stages of the program.

We believe that the contract effort has resulted in a new class of high performance adhesives with RA 59 as the first product of this class, and that, with further development, RA 59 type resin should fulfill the objectives of application to Gr/Pi composite retrofit components for the orbiter. It is recommended that the current effort be continued to resolve the questions previously raised and to characterize and optimize adhesives of this type for Project CASTS application wherever wide range temperature capability is needed.

The report contains a brief description of the experimental work conducted at the Science Center and a detailed discussion of verification testing by the Satellite Systems Division.

3. REQUIREMENTS

TASK I. ESTABLISH CRITERIA FOR SELECTION

The adhesive must serve as a satisfactory bond between the RSI tiles and Gr/PI substrate over the full environmental and operational regimes of the orbiter. Figures 1 and 2 along with Table I of the SOW define Shuttle orbiter design criteria. While these requirements are well defined, the actual conditions existing at the bondline and at each interface are not well known. In addition, processing and refurbishment must be considered. However, except for temperature capability inherent to the characteristics of the material, the suitability of the adhesive, RTV 560, to perform as a bond agent for TPS tiles has been determined primarily by empirical methods. Based on experience with RTV 560, the following criteria were selected as the basis for evaluating a new, higher performance adhesive:

1. Minimum cohesive strength of 25 $\pm$ 5 psi at 650 F (343C), 4 of 5 specimens, 2 batches.
2. When tested with tiles, all failures should be within the tile, not at the bondline (adhesive) or within the adhesive (cohesive). For a valid test, failure within the tile must equal 8 psi or above
3. Thermal cycle tile/composite bond twice, allowing time to stabilize followed by RT tests consistent with (2) above. Cycle limits will be -300 to 700F as a goal with -250 to 600F required.

4. Manufacturing compatibility

   Minimum potlife: 30 minutes

   Maximum cure time at RT: 24 hours

   Mastic-like consistency

   Removable without damaging substrate

5. Environmental Exposure

   Humidity

   Salt spray

   Fungus

   Fuel

   Ultraviolet

   Acoustic

The capability of withstanding environmental exposure should be based on judgment considering the generic properties of the adhesive.

TASK II. SELECTION OF BOND EVALUATION TECHNIQUES

The following evaluation methods were selected which were consistent with the scope of work while providing reasonable confidence that each of the requirements had been addressed:

- Lap Shear Testing - Selected as the simplest method of determining basic strength of the material and bond and determining failure mode.
- Flatwise Tensile Testing - More difficult and expensive than lap shear. However, FWT tests provide a better simulation of actual conditions and permit testing with tile as well as structural adherends. A variety of conditions were imposed on lap shear and FWT specimens to cover both thermal steady-state and cycling situations over the range -300F to +700F.

- o Direct-Bond/Cold Soak - 6" x 6" tile specimens were bonded directly to Gr/Pi honeycomb panel and subjected to progressively lower soak temperatures - as low as -300°F - to determine if failure occurred due to differential contraction. Cold soak was followed by full tile tensile testing (Process Quality Validation, PQV).
- o Cold Soak - Tile specimens were coated with adhesive and soaked at temperatures as low as -300°F to determine if failure occurred due to differential contraction.
- o Refurbishment - Capability of removing the test adhesive from the Gr/Pi substrate without damage using simple mechanical methods and/or solvents as with the RTV 560 was to be demonstrated.
- o Processing - Capability of mixing, preparing, and applying the adhesive in reasonable quantities without imposing difficult constraints or controls was to be demonstrated.

Other methods of test and analysis which were to be used but for which criteria for selection were not established included:

- o Differential colorimetry (DSC)
- o Thermo-gravimetric analysis (TGA)
- o Thermo-mechanical analysis (TMA)
- o IR spectrographic analysis
- o Microscopic analysis
- o Peel tests

TASK III. ADHESIVE SCREENING

Screening was accomplished by in-house effort prior to the starting funded work under the contract to the point of concentrating on the RTV 560 - glass resin mixtures. Results of preliminary tests, presented to NASA during

the third month, resulted in approval to proceed with these mixtures exclusively and to eliminate any further consideration of using a strain isolation pad (SIP).

TASK IV. ADHESIVE SYSTEM CHARACTERIZATION

Specimens were tested and analyzed using all of the methods selected for evaluation as defined in Task II.

TASK V. PROCUREMENT SPECIFICATION OUTLINE

An outline specification was prepared for procurement, including quality control provisions.

TASK VI. PROCESS SPECIFICATION OUTLINE

An outline specification was prepared for processing the adhesive system.

4. SCREENING AND SELECTION

Under a prior IRD program within Space Division, as reported in Reference 1, extensive screening of materials was conducted which identified two candidates with a potential for room temperature curing. These were polyphenylquinoxalenes (PPQ) and sesquisiloxanes (SQS). Experimental effort at the Science Center developed the capability for processing and solvent curing PPQ. However, the resin failed to meet criteria subsequently developed (Section 3.0) from the standpoints of both processing and toxicity. SQS, while nontoxic and capable of RT curing had insufficient "body" and, upon curing, was too friable to permit consideration as an RSI bonding agent. Yet of all the materials identified, SQS-type materials alone appeared to offer the potential for development to meet the full range of requirements. In an attempt to combine the best properties of SQS and RTV, mixtures of the two resins were prepared and evaluated as the initial work conducted under the contract program. Experimental work involving these mixtures is summarized in the following section and discussed in some detail in Appendices A and B.

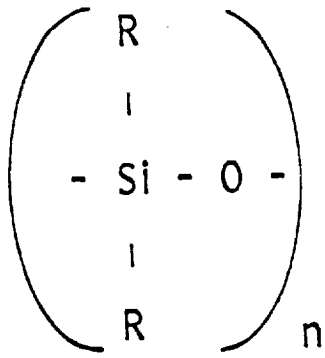
5. EXPERIMENTAL TESTS

SQS polymers are of the general type described in Reference 3. Their planer structure, as shown in Figure 1, provides thermal stability without cross-linking. The result predictably is the achievement of higher strength once sufficient temperature has been achieved to result in cross-linking. The difficulty is in providing required characteristics at RT without destroying the inherent ability to perform at high temperature.

For experimental tests, small quantities of three SQS resins were obtained from Owens-Illinois. Designated by OI as types 100, 650 and 908 glass resins (GR), these had slightly differing formulations as indicated in Figure 1 (i.e., methyl, phenyl, etc.). Glass resins, received in flake form, were ground, dissolved in acetone and mixed with RTV 560. The most workable mixtures were initially obtained with 30.5% GR (glass resin) 650 in RTV 560. Experimental testing was concentrated primarily on this mixture and variations thereof. RTV 560 was used as a control throughout. Results of these tests indicated the following:

- The new mixture and the control have comparable strength at RT.
- The mixture has 2 x control strength at 600F and 4 x control at 700F.
- Similar results were obtained with and without SIP.
- The use of primer increased performance at RT but not at high temperature.
- Excursion to high temperature significantly increased subsequent bond strength of the mixture but not of the control.
- Mixtures of GR in RTV 566 (a highly refined RTV 560) showed no different results from those with RTV 560.

RTV 560 - SILICONE



GLASS RESIN (OWENS ILLINOIS) - SESQUISILOXANE

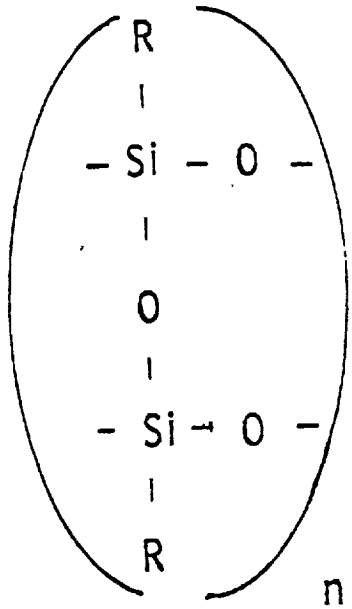


Figure 1. SQS Molecular Structure

Test results, shown in Appendix A, were presented to representatives of the CASTS Program Management and members of the Materials Division on March 7, 1978. At that time, based on results to date, authorization was given to proceed with the remainder of the program and to emphasize the direct bond application, eliminating SIP from future test specimens.

Subsequent to the March meeting problems were encountered in which mixtures of RTV 560 and GR 650 were often lumpy and difficult to bond. Upon recommendation by OI a change was made to type 908 as being a more stable formulation with longer shelf life. Additional experimental work led to selection of a mixture of 25% GR 908 in RTV 560 as the "test" adhesive. This was subsequently designated RA 59.

Experimental testing continued to evaluate RA 59 at temperatures to 700F. However test specimens and conditions were modified in an attempt to better simulate the orbiter application. RSI tile specimens were cut from Li 900 material obtained as manufacturing rejects. These were 1.5 inch diameter cylinders one-half inch thick. Tiles were bonded to Gr/PI adherends which had previously been sandblasted or scrubbed using "Bear Tex" to pass a water-break test.

Flatwise tensile test (FWT) specimens were then fabricated. Early tests, conducted at soak temperatures of up to 700F in an Instron, were made in which the entire FWT specimen was subjected to the test condition. This procedure proved unsuitable for thermal cycling however due to failure of the HT424 adhesive at the brass block - Gr/PI bondline. Instead, cycling (temperature excursion) was performed on the tile - Gr/PI specimen then brass blocks were bonded and FWT tests conducted at room temperature.

The photograph of Figures 2 and 3 show typical failures occuring subsequent to excursion testing. Failure of the RA 59-bonded specimens was almost invariably within the tile at tensile loads $\geq$ 12 psi. Failure of RTV 560 control specimens almost invariably exhibited some percentage of adhesive failure at the tile interface.

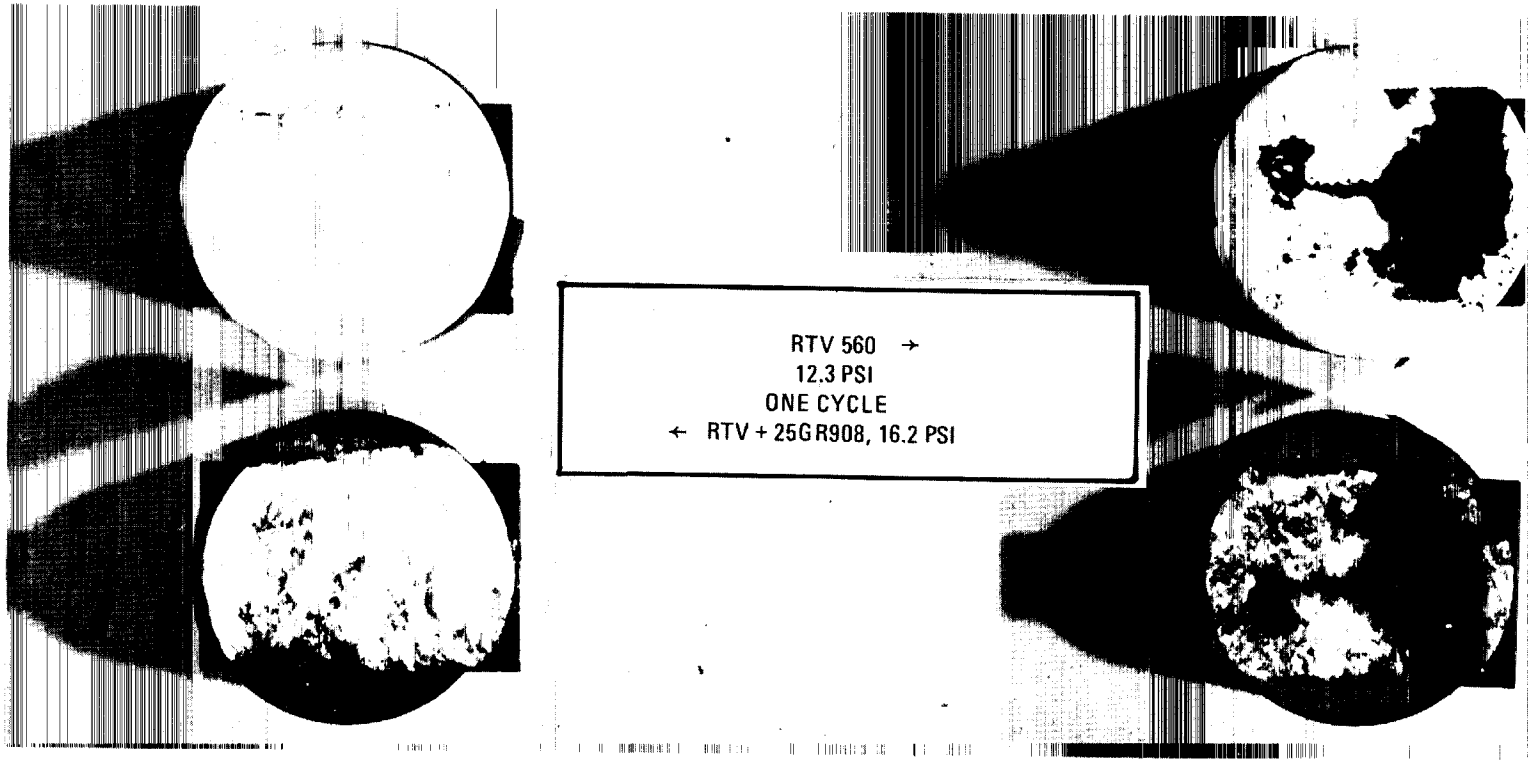


Figure 2. Comparison of FWT Test Specimen Failures

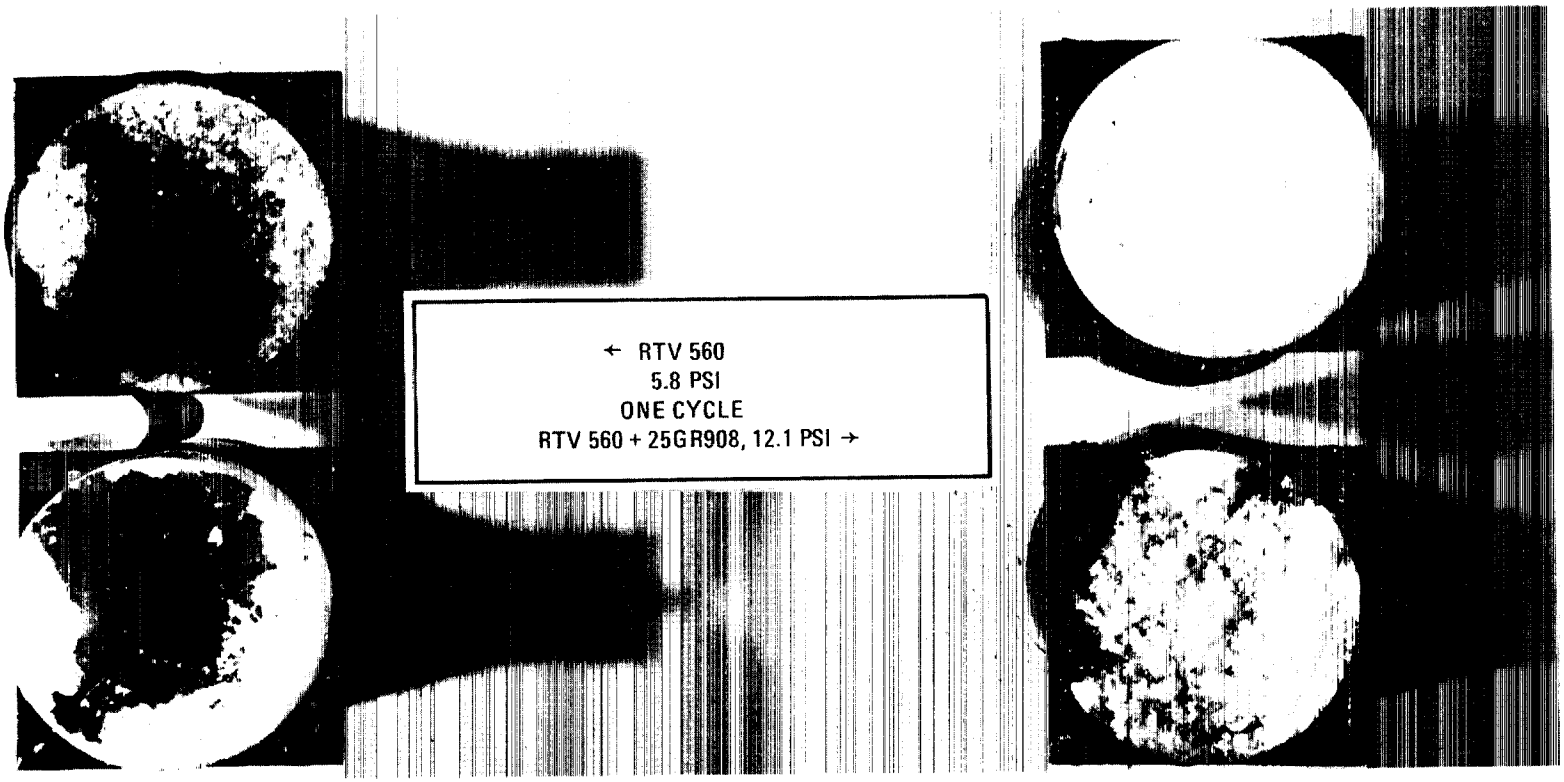


Figure 3. Comparison of FWT Test Specimen Failures

6. VERIFICATION TESTING

6.1 INTRODUCTION

The laboratory development and verification test program was to evaluate a modified RTV 560 with improved thermal stability at 700F. As previously discussed development and initial testing had been accomplished at the Science Center prior to initiation of verification testing.

The formulation for modifying RTV 560 silicone adhesive with a solid silicone resin to increase its thermal stability was provided to the Space System Group Materials and Processes Laboratory by the Rockwell International Science Center. This material has tentatively been identified as RA 59. Goals of the program were: to develop scale-up and processing techniques for producing and using the adhesive; to perform thermo-chemical and mechanical property tests; and to demonstrate the validity of the material through full scale tile bonding and thermal cycling. In all testing, RTV 560 was to be included as a control material.

Procedures for preparing RA 59 in one pound quantities were developed which can be used as a basis for mixing larger amounts. Thermal cycling of RSI tiles bonded directly to a graphite/polyimide honeycomb structure resulted in acceptable performance by both adhesive materials. Thermo - chemical testing of RA 59 gave predictable results based on known characteristics for each component and demonstrated an improvement in thermal stability for RA 59 over RTV 560.

During mechanical property testing, which consisted of tensile adhesive tests, several questions were raised as data were accumulated. Initial data indicated that increasing exposure time at 700F tended to increase the strength of RA 59 while it degraded the properties of RTV 560. Additional testing at various temperatures up to 700F didn't show as distinct an improve-

ment over RTV 560 and subsequently, the thermo-chemical analysis was broadened to investigate the RA 59 more thoroughly. Questions raised during this effort which should be answered prior to initiating a qualification program included: 1) is there a chemical reaction among the Si containing components of the adhesive system; 2) does the catalyst used act selectively with RTV 560 or randomly with each silicone; 3) would a separate catalyst for each component improve silicone interaction and system performance; 4) what is the primary cause of the wide range of tensile adhesive values at 700F.

6.2 PURPOSE

The purpose of this laboratory phase of the program was to verify that the adhesive formulation provided by Rockwell International Science Center personnel is a valid material for consideration in bonding RSI tile to graphite composite structures. This goal was to be accomplished by performing the following investigations:

1. Develop processing techniques for the preparation of large batches of RA 59 adhesive compound and evaluate working characteristics.
2. Determine the thermo-chemical properties of the cured adhesive formulation.
3. Determine the adhesive properties of RA 59 after thermal exposure.
4. Evaluate the performance of RA 59 when used to join RSI tile to polyimide/graphite composite honeycomb panel during exposure to temperatures over the range -300 to 700F.
5. Compare each of the above investigations with RTV 560 baseline adhesive.

6.3 CONCLUSIONS

In general, it can be concluded from the results of this investigation that RA 59 is comparable to RTV 560 in all significant aspects and has superior elevated temperature adhesive properties after exposure to temperatures at or above 600F. Specific conclusions include:

1. Techniques for mixing Owens-Illinois 908 silicone resin into RTV 560 on a large scale were developed and demonstrated in the preparation of three separate batches of RA 59 in quantities up to one pound.
2. Thermo-gravimetric analysis data indicate that cured RA 59 and RTV 560 demonstrate similar thermo-chemical characteristics at elevated temperatures.
3. In general, increasing exposure time at 700F increases the strength of RA 59 while degrading the properties of RTV 560.
4. These were no apparent cracks in RSI tile bonded directly to a graphite composite honeycomb skin with RA 59 and RTV 560 after cycling to -300F. A layer of each adhesive applied to tile surface but unrestrained by bonding to graphite composite laminate resulted in tile cracking during thermal cycling to -300F.

6.4 PROCEDURE AND RESULTS

The laboratory effort performed during this program consisted of three separate subtasks: (1) RA 59 preparation and process development; (2) mechanical and physical property determination; and (3) performance demonstration. Each of these are discussed in subsequent paragraphs.

6.4.1 RA 59 Preparation and Process Development

Initial small batches of adhesive mixture were prepared by dissolving Owens-Illinois 908 resin in acetone, adding small quantities of that mixture to RTV 560, stirring until a smooth paste is obtained and, finally, allowing acetone to evaporate from the compound. Hand mix time was approximately 30 minutes and acetone evaporation required up to 17 hours.

For large batch scale-up preparation of the RA 59 adhesive mixture, several automated techniques were investigated.

1. A mixture of 3.3 grams OI 908 and 18 grams acetone was added to 10 grams of RTV 560 in a pint can and agitated on a Red Devil paint shaker for one hour. There was no mixing of the 908/acetone solution with the RTV 560.
2. Twenty five grams of acetone were combined with 75 grams RTV 560 and agitated 15 minutes on paint shaker. In a separate container 25 grams of 908 were dissolved in 50 grams of acetone. The two mixtures were poured into a pint can 1/4 full of clean, dry, ceramic pebbles and agitated on paint shaker for one hour. There was no mixing of 908/acetone with RTV 560.
3. A mixture of 66 grams OI 908 resin and 154 grams acetone was added incrementally to 150 grams of RTV 560 in a pebble mill container 1/3 full of ceramic pebbles. After five hours of rotation, mixing of all components was incomplete.
4. Based on failures of the above techniques, it was concluded that a means of duplicating hand mixing using mechanical equipment should be explored. Therefore, a metal-blade stirrer was attached to an air-powered drill motor as shown in Figure 4. The motor was set at 180 RPM with blade in RTV 560 and 908/acetone mixture was added in 5-10 cc quantities at 10 minute intervals. Three batches of RA 59 material ranging in size from 266 to 532 grams were successfully prepared using this technique.

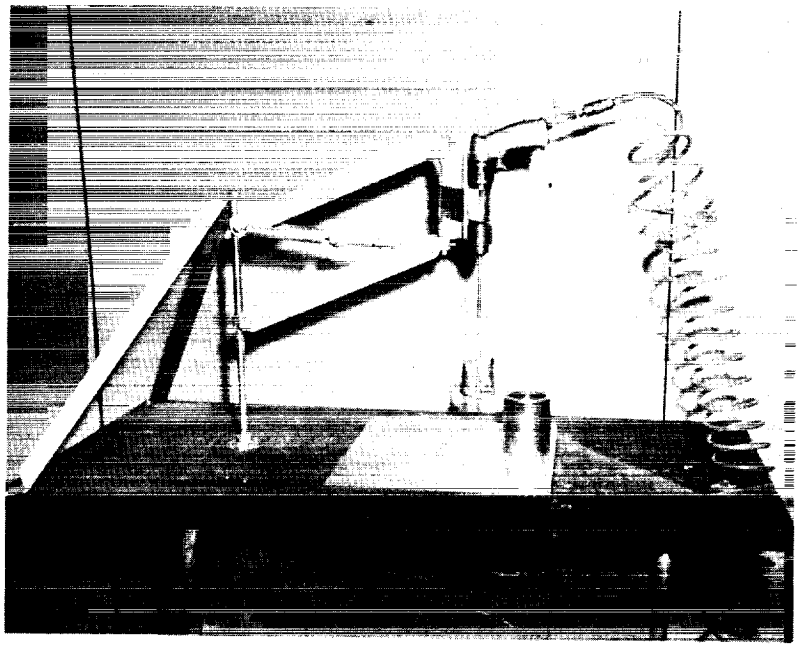


Figure 4. Mechanical Mixer

In order to evaluate RA 59 as an adhesive, three processing questions were investigated: (1) the optimum percentage of di-butyl tin di-laurate catalyst (Thermalite, T-12) required to provide optimum mechanical properties and processing time; (2) cure time required for maximum properties; and (3) need for a primer for bonding to materials other than silicone or silica.

These questions were investigated by means of tensile lap shear adhesive tests, Shore A hardness measurements, peel adhesive tests and thermo-gravimetric analysis. RA 59 was prepared from RTV 560 batch 719 and OI 908 batch 51666 using the power driven mixer technique described above. The RA-59 was catalized with 0.5%, 1.0% and 1.5% T-12 and used to prepare the following specimens:

Lap Shear - per Federal Test Method Standard MMM-A-132 using 0.050-inch steel adherends (See Figures 5 and 6) with and without SE4155 primer, minimum 1.5 psi dead weight pressure with 0.010-inch wire in bond line to control thickness.

Hardness - Material cast in aluminum cup resulting in specimen 1/4-inch thick by 2 inches in diameter.

Peel - T-peel specimen per ASTM using 0.020-inch aluminum adherends, with and without SE4155 primer, and minimum 1.5 psi dead weight pressure.

Thermo-gravimetric Analysis - catalyzed RA 59 de-gassed under vacuum and cast 1. x 1. x 0.187-inch thick specimens. In all cases control specimens were prepared from the same batch of RTV 560 and T-12 catalyst.

Results of the process variable investigation indicated that: (1) 1.0% catalyst concentration (of the RA 59 mixture which is 1.3% of the RTV 560 present) is optimum for adhesive properties; and (2) use of SE4155 silicone primer substantially improves adhesion to metallic substrates. In addition, maximum cure of RA 59 with 1% catalyst is achieved within 15 days. However, 90% cure (as measured by hardness) occurs within three days and bonded components could be handled within 48 hours.

Data to substantiate these conclusions are summarized in Figures 7 through 9 and Table 1. Figure 7 gives the results of lap shear testing of specimens at room temperature after exposure to 700F for four hours. Cure time as indicated by Shore A hardness is shown in Table 1 and Figure 8. The effect of thermal exposure in air as indicated by weight loss for RA 59 with varied catalyst concentration is given in Figure 9. T-peel testing was found to be an invalid method of quantitatively determining the effect on

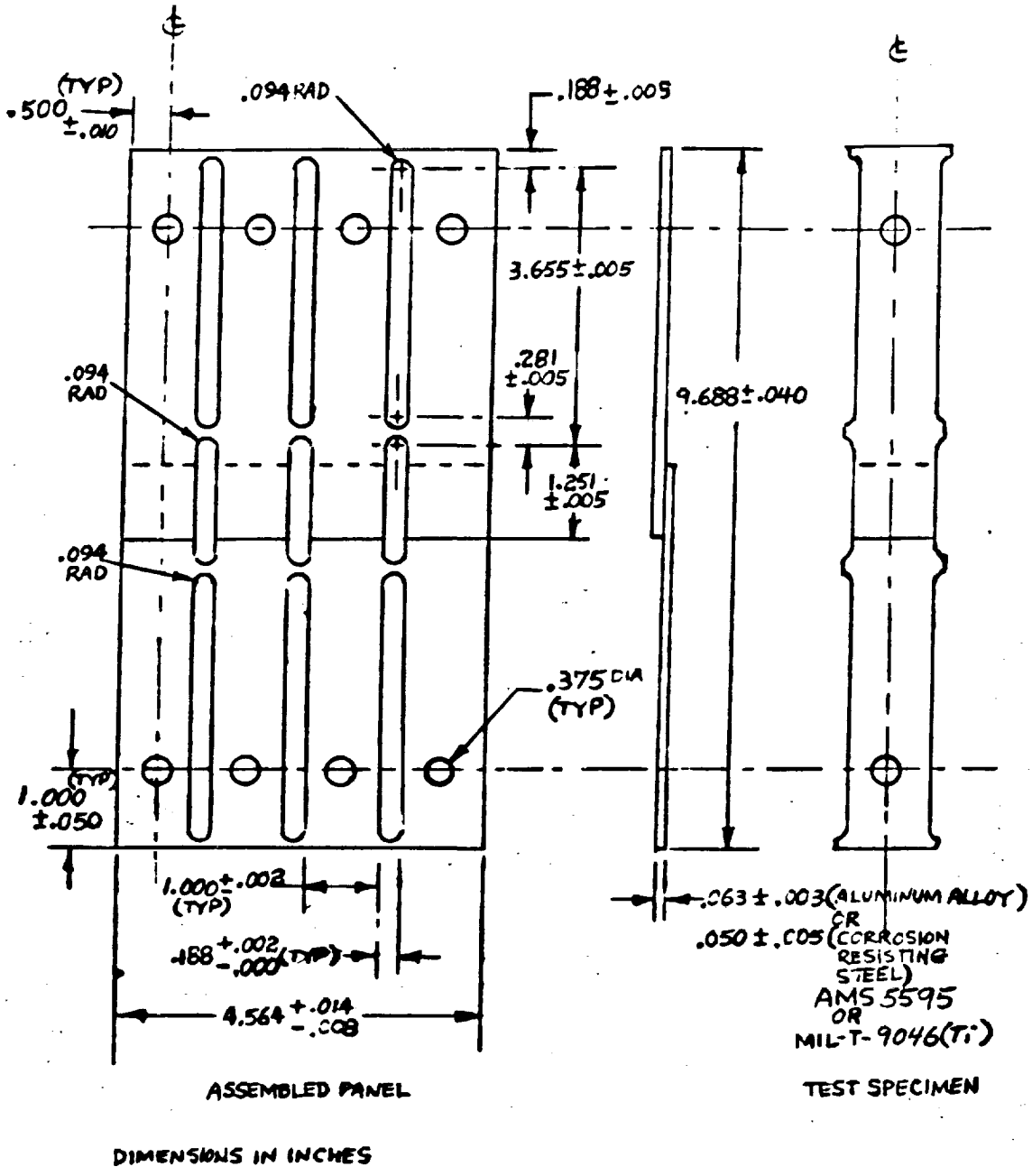
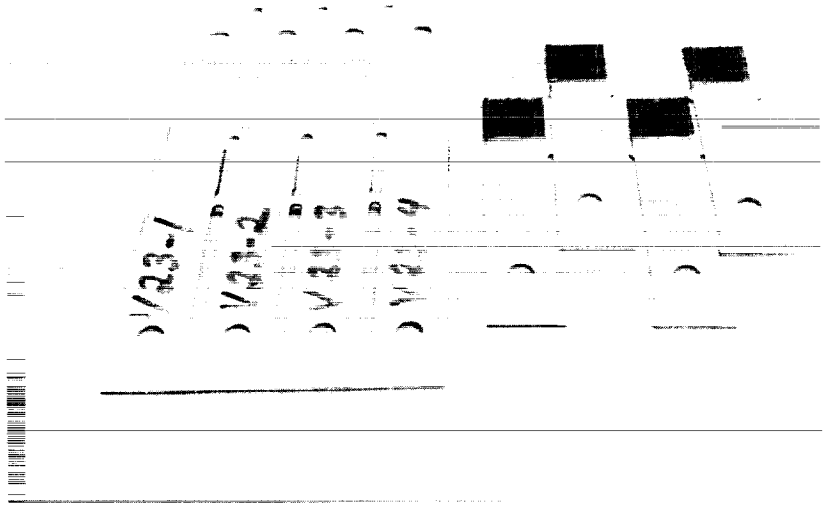


Figure 5. Tensile Lap Shear Panel and Specimens



Bonded Configuration Failed Specimen Halves

Figure 6. Tensile Lap Shear Panel and Specimen

adhesion of the use of primer. This was because the modulus of aluminum, even in the "S" condition, was too high to permit proper bending of the specimen before adhesive failure occurred at the bond line. Instead, the recommendation to use primer is based on qualitative information such as the failure of un-primed lap shear specimens during handling and the total adhesive failure mode (no adhesive on metal surface) for all peel specimens and most lap shear specimens bonded without primer. Based on the data produced during this process investigation phase of the program, all subsequent specimens were made from RA 59 catalyzed with 1.0% T-12 and primer was used on all surfaces which were not silicone or silica.

TENSILE LAP SHEAR STRENGTH VS CATALYST CONCENTRATION

TESTED AT R.T. AFTER 4 HOURS AT 700F

• = RA 59 WITH PRIMER

0 = RA 59 NO PRIMER

+ = RTV 560 PRIMER

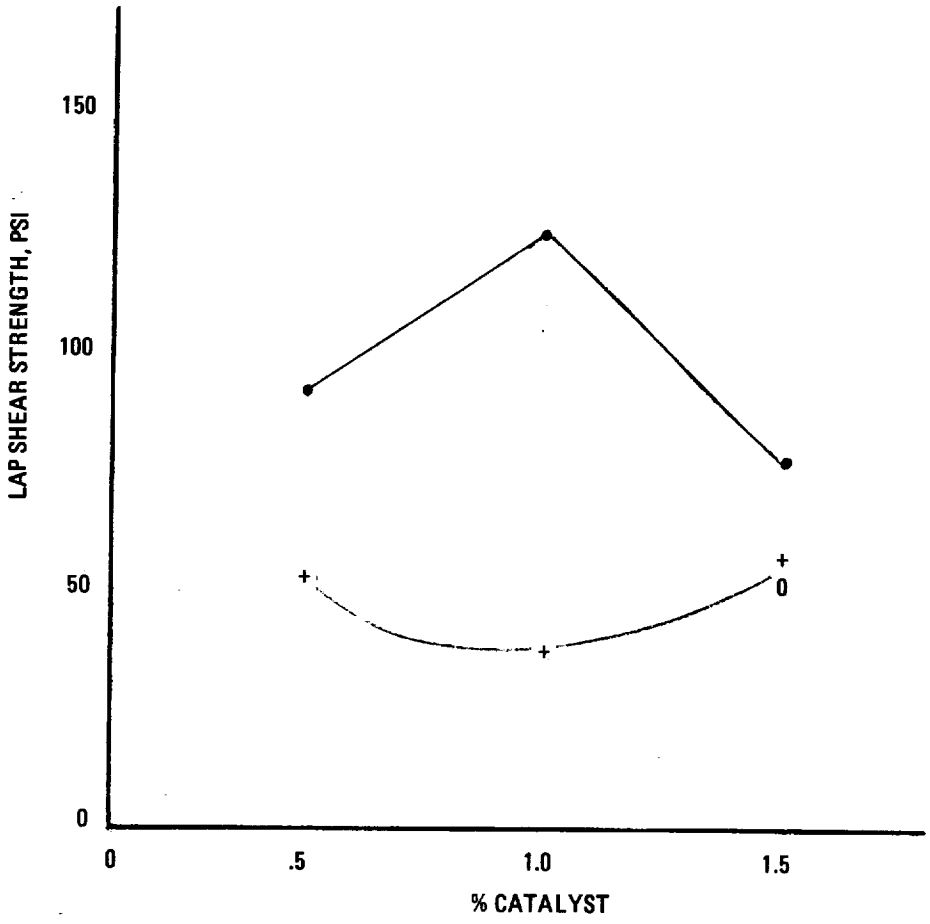


Figure 7. Tensile Lap Shear Strength Versus Catalyst Concentration

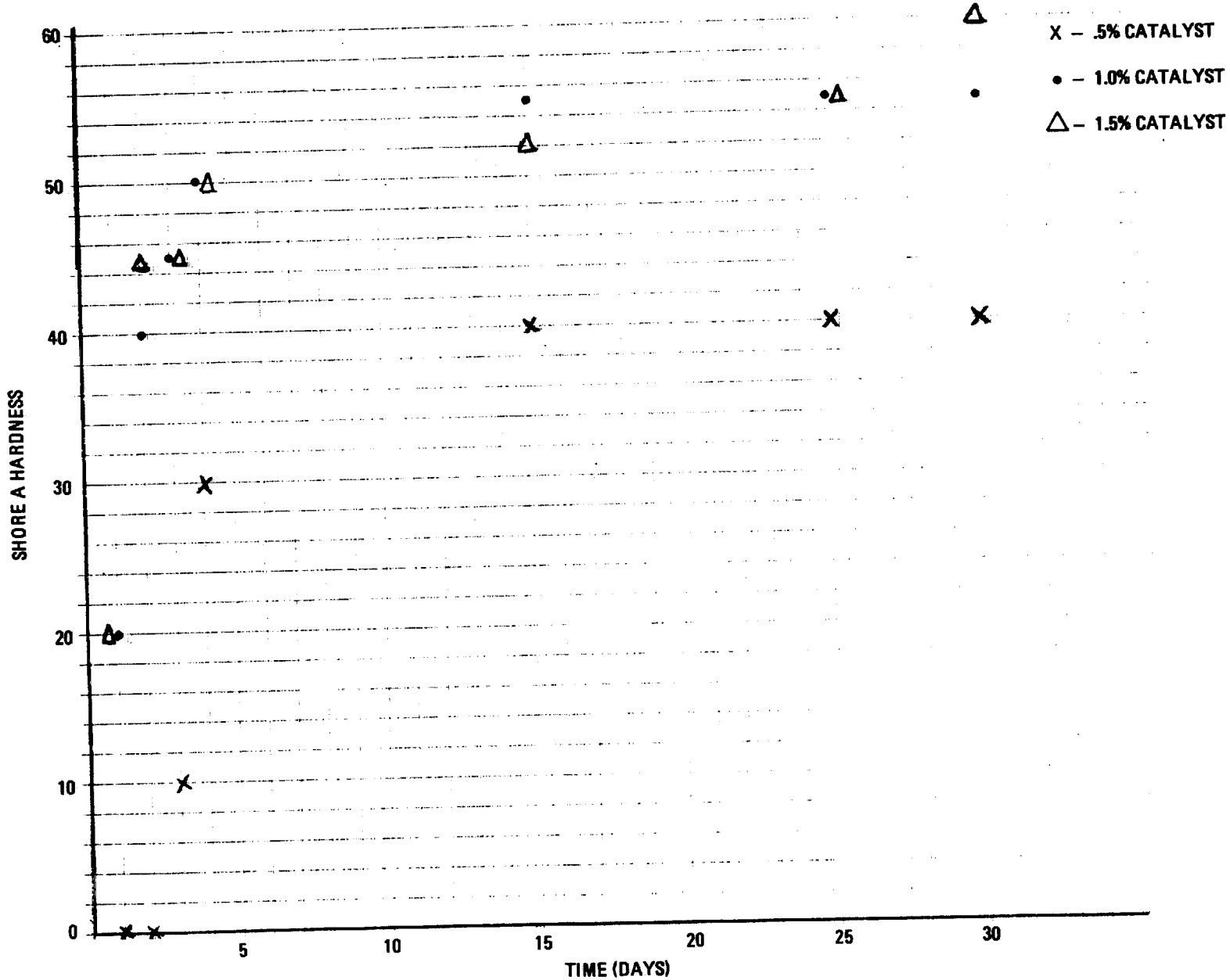


Figure 8. Cure Time for RA 59

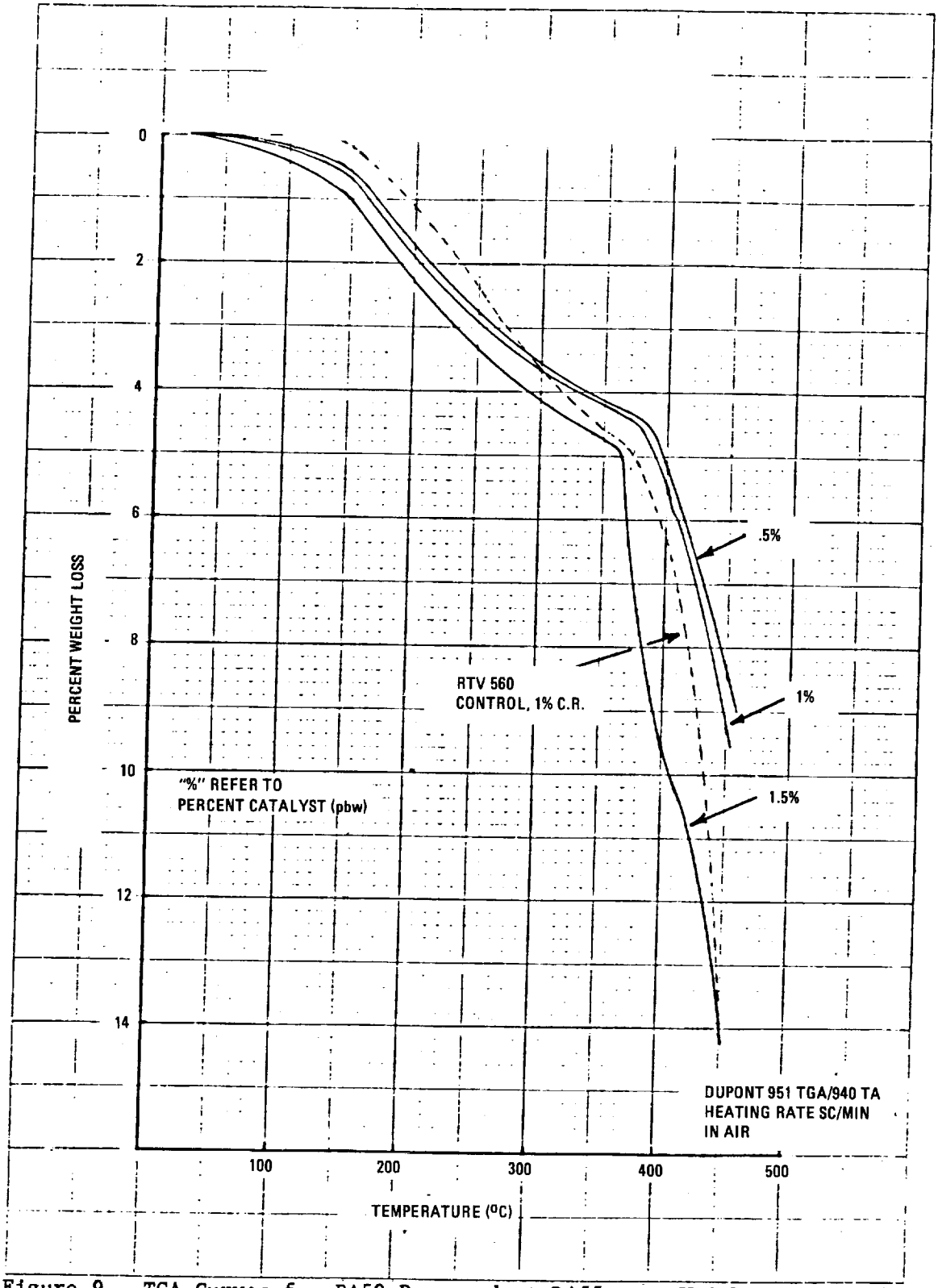


Figure 9. TGA Curves for RA59 Prepared at Different Weight Percentages of Curing Agent

Table 1. Shore A Hardness Versus Catalyst Concentration Versus Time

Material	Catalyst Concentration (%)	Days						
		1	2	3	4	15	25	30
RA-59	.5	Soft	Soft	10	30	40	40	40
RTV-560	.5	50	50	50	50	50	50	50
RA-59	1.0	20	40	45	50	55	55	55
RTV-560	1.0	50	50	50	50	55	55	55
RA-59	1.5	20	45	45	50	52	55	60
RTV-560	1.5	50	50	50	50	54	54	55

During the preparation, use and evaluation of RA 59, the effect of additional material and process variables on mechanical and physical properties was studied. Variables noted and/or evaluated included physical appearance of RA 59 compound prior to catalyst addition, RA 59 batch variation relative to RTV 560 batch and shelf life of RA 59 mixture. Evaluation of variables was primarily based on lap shear testing, however, visual observation and TMA testing was also used.

It was noted that smoothness of RA 59 mix varied between batches of the RTV 560 component as shown by the following material combinations:

Supplier's Batch No.			
RTV 560	OI 908	Date Prepared	RA 59 Mixture Appearance
719	51600	7/12	Smooth-no lumps or graininess
719	51666	7/31	Smooth-no lumps or graininess
731	51666	8/4	Granular, sand filled
719	51666	9/15	Smooth-no lumps or graininess

(This condition was also noticed at the Science Center during the experimental phase of the program.) Lap shear test data were tabulated to correlate RA 59, RTV 560 and OI 908 batch number and RA 59 shelf life with lap shear strength. These data which are presented in Table 2 show no trend among these variables and therefore, may not be significant relative to RA 59 performance. However, the spread in tensile data noted in a subsequent section of this report (Figure 14) may result from and be controllable by material or processing variables. Areas for further investigation include iron oxide particle size control, OI 908 resin variables and effect of time after catalyzation.

Table 2. Tensile Lap Shear Strength RA 59

Spec.	RA-59 Batch	RTV 560 Batch	GR 908 Batch	Fab Date	Test Temp/Conditioning Temp (psi)*					
					RT/RT	RT/700	700/RT	700/700	RT/175	700/175
1-2	50 GR	719	51666	7/24		126				
V-2	2	731	"	8/9		128				
V24	2	"	"	9/1		54				
V6	2	"	"	8/9				59		
V25	2	"	"	9/1				34		
V9	2	"	"	8/9	200					
V11	2	"	"	8/9	245					
V16	2	"	"	8/9	238					
V17	2	"	"	8/9	220					
V26	2	"	"	9/1	360					
V27	2	"	"	9/1			18			
V36	2	"	"	10/3		50				
V37	2	"	"	10/3				23		
V38	3	719	"	10/3		80				
V39	3	719	"	10/3				38		
V42	2	731	"	10/3	190					
V46	3	719	"	10/3	320					
V41	2	731	"	10/3				15		15
V45	3	719	"	10/3				15		15
V40	2	731	"	10/3					394	
V44	3	719	"	10/3					328	

*The first temperature is that at which specimens were tested; the second is that temperature at which specimens were soaked for 4 hours prior to test. All values are an average of four specimens. Range of values is given in Table 3.

6.4.2 Mechanical and Physical Property Determination

The second subtask of the laboratory program consisted of examining the effect of thermal exposure, humidity and R.T. aging on adhesion as measured by tensile tests. In addition, thermo-physical properties of the RA 59 adhesive as well as the OI 908 resin were studied.

Since 700F is the temperature at which thermal degradation becomes severe as shown by TGA (Figure 9), and since bond line temperature could approach that temperature during Orbiter flight, the effect on adhesion versus time at 700F was determined. Steel finger specimens per FTMS MMM-A-132 were primed with SE 4155 and bonded with RA 59 using 0.010-inch wire for thickness control and 2.0 + 0.5 psi vacuum pressure. RTV 560 specimens were prepared concurrently as control. Figure 10 gives the results of this study which shows a direct correlation between lap shear strength and exposure time at 700F up to eight hours. Cohesive failure (failure within the adhesive) occurred in all specimens.

The effect of humidity and room temperature aging on lap shear strength is under study with preliminary data given in Figure 11. Specimens were fabricated with those described in the preceeding paragraph. No effect was noticed through two months aging.

Adhesion to polyimide/graphite composite laminate was evaluated by means of double lap-strap tensile shear specimen shown in Figure 12. Materials and cure procedure were the same as those described above except that wire was not used for thickness control.

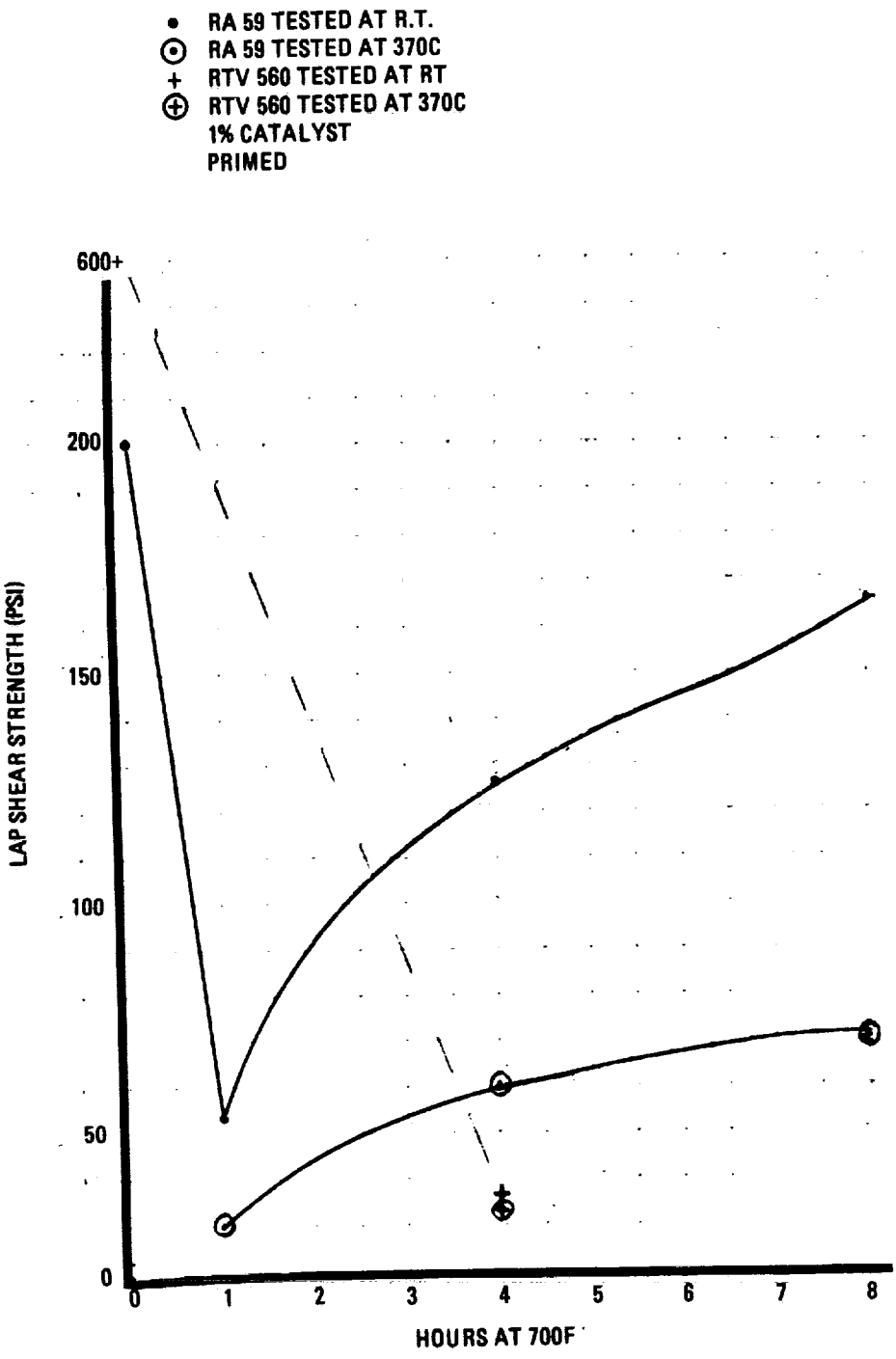


Figure 10. Shear Strength Versus Temperature Exposure

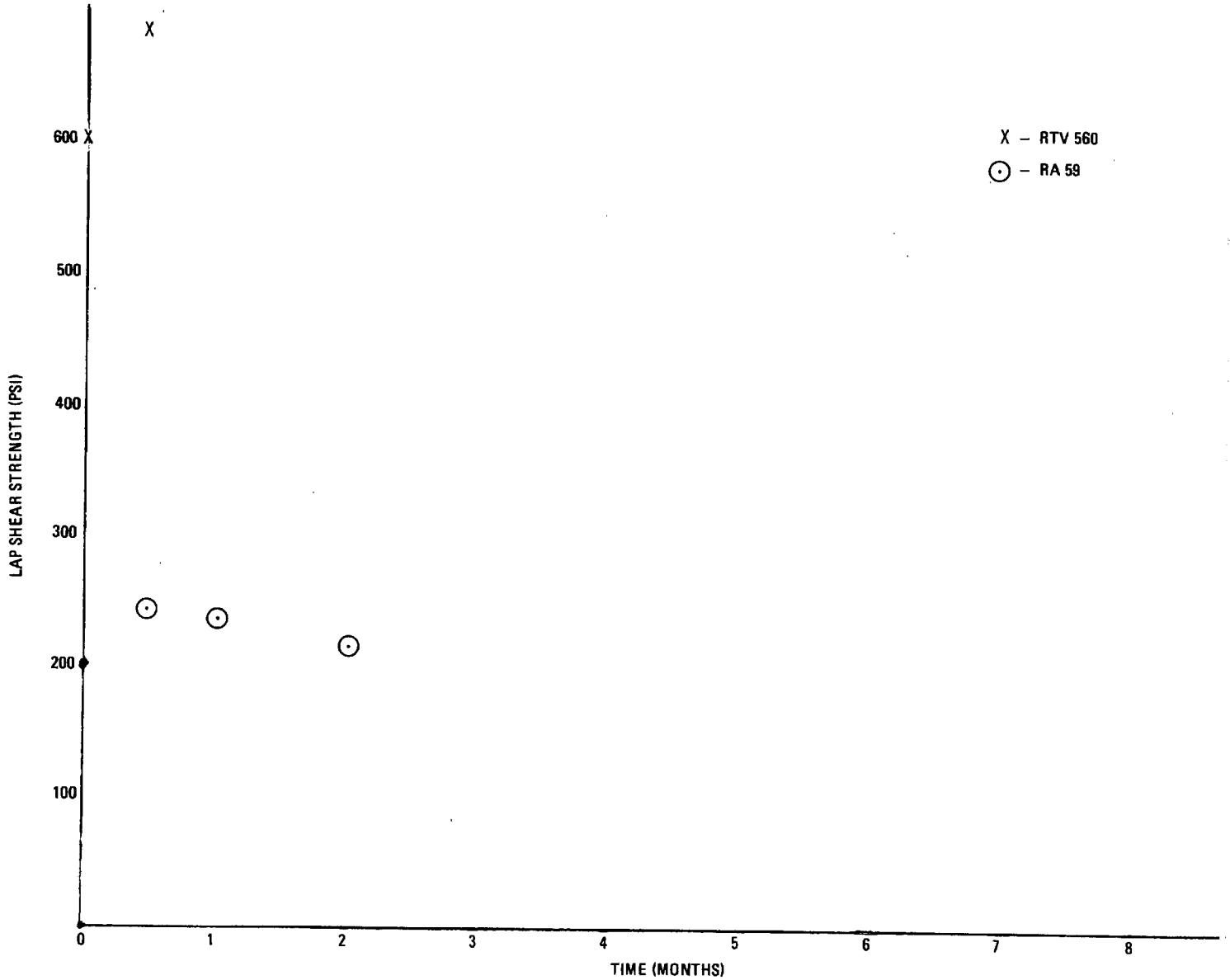


Figure 11. Lap Shear Strength Versus Aging (95% Relative Humidity for Two Weeks at RT)

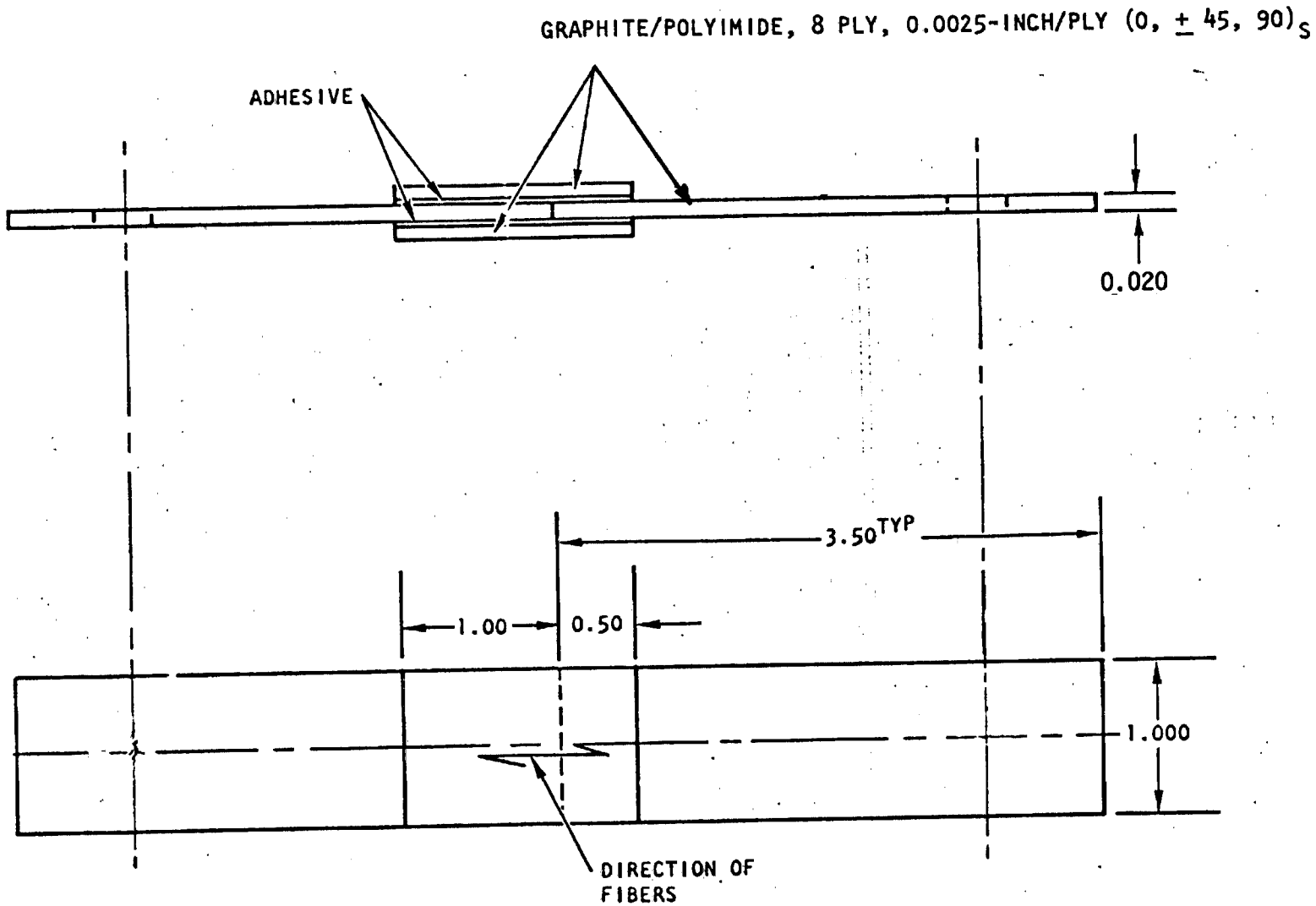


Figure 12. Double Lap Strap Shear Specimen

Test results shown in Figure 13 indicate much lower room temperature values than with steel adherends for both RA 59 and RTV 650. Since failure mode was cohesive it is felt that the lower values are a result of specimen configuration rather than adhesion to graphite. In addition, the 15 psi average at 700F for RA 59 with no prior conditioning correlates with 18 psi average for steel adherends tested under the same conditions.

Thermo-physical property tests indicated that molecular changes occurred within RA-59 at temperatures as low as 140F. A group of lap shear specimens was prepared for testing after exposure to temperatures below 700F in order to determine if low temperature post cure can provide high temperature stability. In order to give some indication of shelf life of uncatalyzed mixture the same batch of RA 59 and processing parameters used for the 700F study were used for these specimens. Testing was performed at room temperature and at 175F 300F, 600F and 700F with prior conditioning for four hours at those temperatures. Because of a reduction in tensile strength at 700F, a new batch (identified as batch 3) of RA 59 was prepared using RTV 560 Batch No. 719 which originally produced the smooth, non-granular mixture discussed above. Batch 3 was a smoother mixture than Batch 2. However, as shown in Table 2 and Figure 14, this factor of smooth vs. grainy RA 59 was not always, or alone, significant in affecting shear strength.

Results of these groups of tests are shown in Figures 14 through 17. Figure 14 gives lap shear strength at room temperature after a four hour exposure at several temperatures. The spread in values at R.T. and 700F is probably caused by unknown and uncontrolled processing variables such as time between mixing and pressure application rather than erratic behavior of the adhesive.

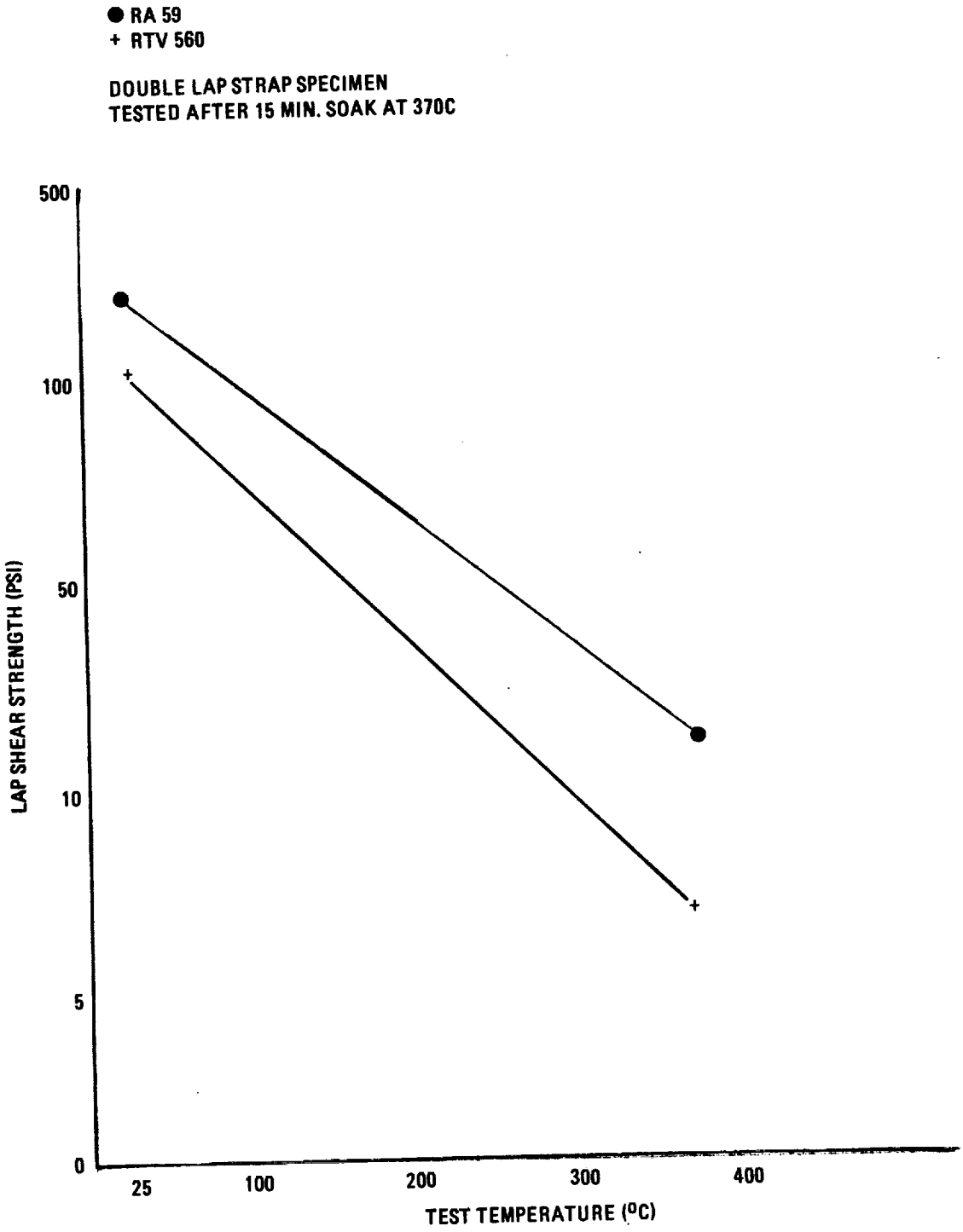


Figure 13. Lap Shear Strength Versus Test Temperature - Graphite Substrate

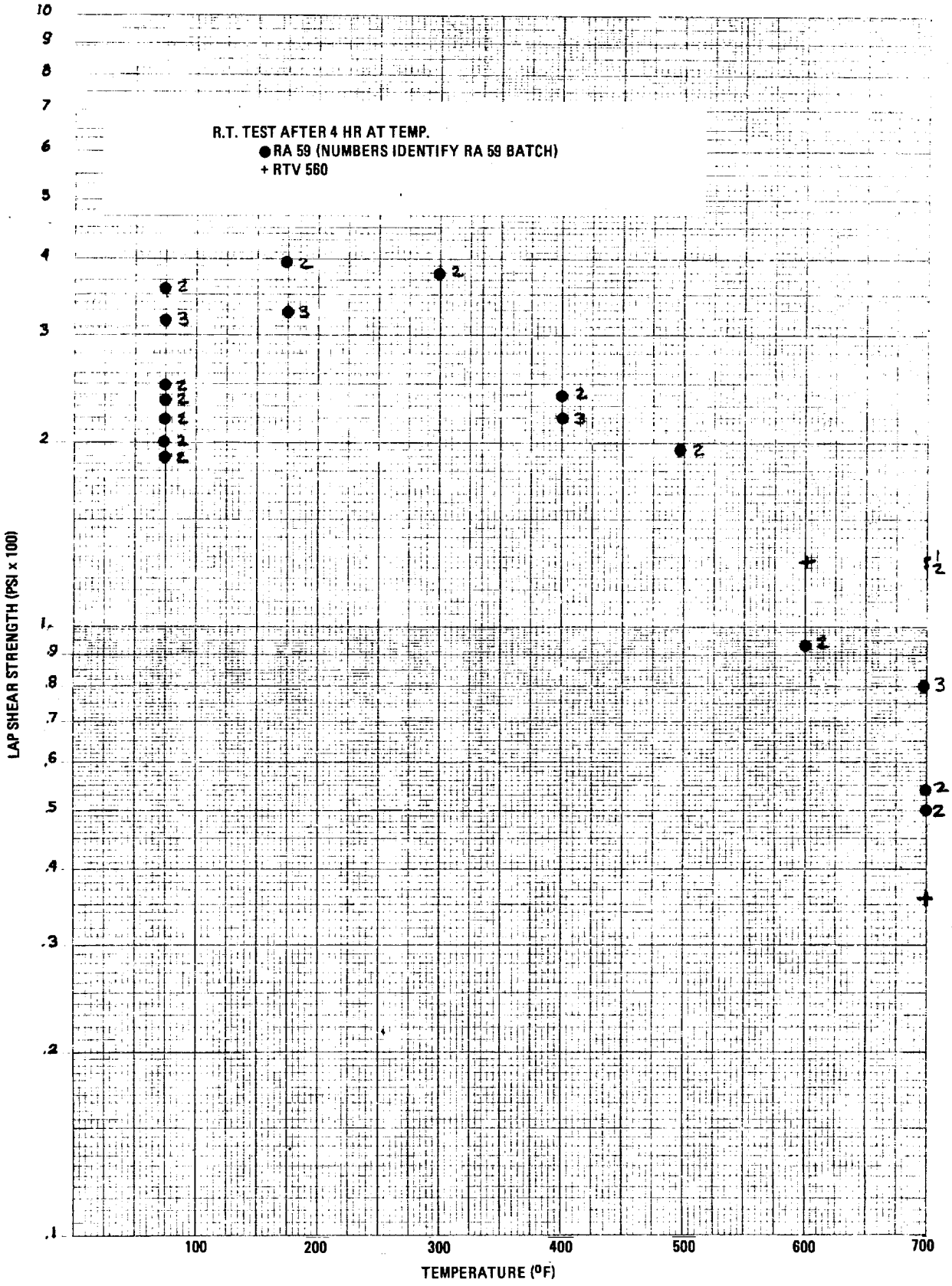


Figure 14. Lap Shear Strength After Thermal Exposure

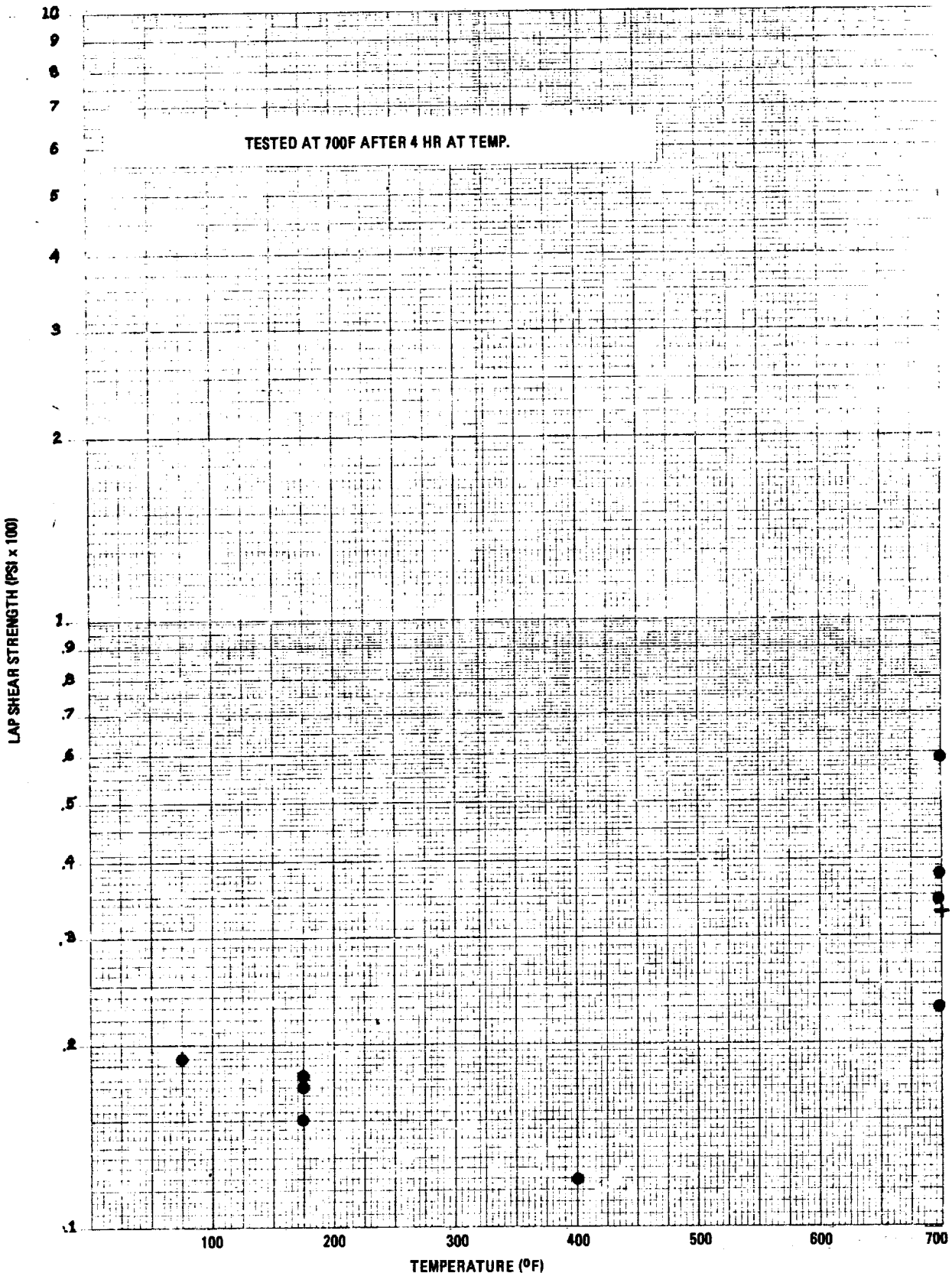


Figure 15. Lap Shear Strength at Temperature After Thermal Exposure

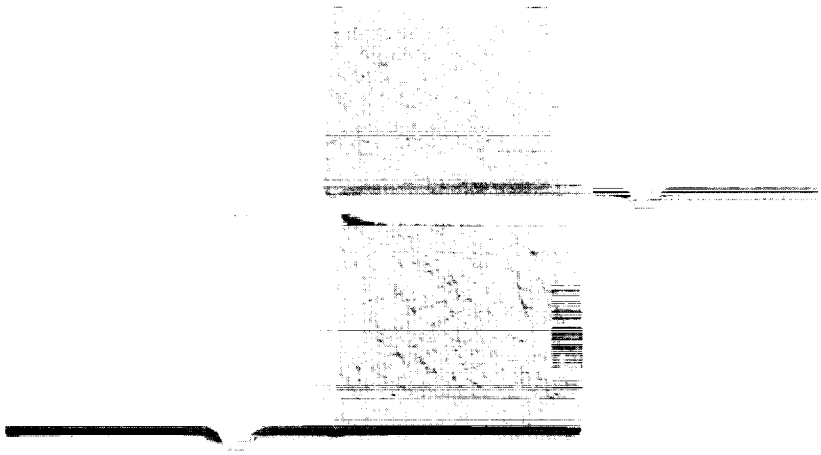


Figure 16. Adhesive After Lap Shear Testing

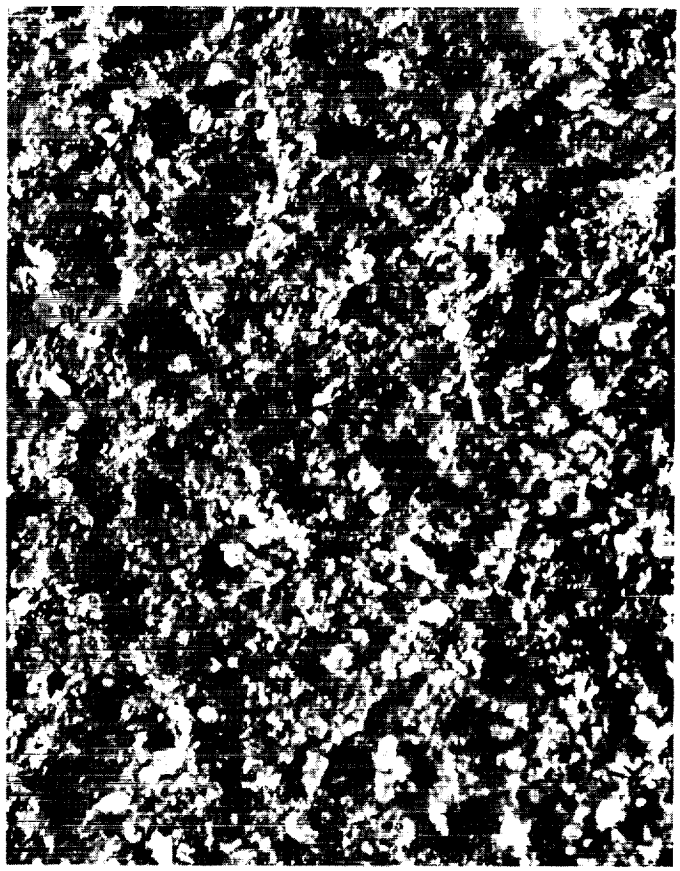


Figure 17. Room Temperature Exposure

This conclusion is based on the narrow range of values from the four specimens represented by each symbol on the graph. Tabulated data in Table 3 demonstrate this point.

Table 3. Lap Shear Test Data Spread

Panel No.	RA 59 Batch No.	Test Temp.	Conditioning Temp. °F	Test Values, Psi		
				High	Low	Average
1-2	1	RT	700	137	120	126
V2	2	RT	700	146	114	128
V24	2	RT	700	57	52	54
V36	2	RT	700	52	49	51
V38	3	RT	700	87	79	81
V9	2	RT	RT	208	190	200
V11	2	RT	RT	267	235	245
V16	2	RT	RT	254	220	238
V17	2	RT	RT	232	188	220
V26	2	RT	RT	380	336	360
V42	2	RT	RT	200	188	190
V46	3	RT	RT	334	300	320

Figure 15 provides data from an investigation of a characteristic of RA 59 probably resulting from the presence of the GR 908 resin in the mixture. Early tests showed an increase in strength at 700F at post cure time at 700F was increased up to 8 hours (refer to Figure 10). Additionally, TGS curves showed a "curing" type reaction as low as 175F. Therefore, the possibility of high strength values at 700F after a lower temperature post cure was investigated with results shown in Figure 15. Although the data indicate that the post-cure temperature must be at least above 400F to provide a completely stable material 700F, adhesive strength is more than adequate after RT cure and no adverse effects of aging have been noted in the absence of a post cure of elevated temperature.

In comparing the exposed adhesive of tested lap shear specimens, as shown in Figure 16, it was noted that the adhesive surface was granular for those specimens which had not been exposed to elevated temperatures while specimens exposed to 370C had a uniform rubber-like appearance. Microscopic examination revealed a heavy concentration of small shiny glass-like globules dispersed throughout the exposed adhesive. From specimens made at the same time with the same adhesive mix but post-cured at 700F, the globules were smaller and significantly fewer. It was apparent that the form and mode of dispersion of the GR 908 in the RTV 560 matrix was changed by application of heat.

This phenomenon was pursued briefly by microscopic examination of failed lap shear specimen adhesive after various exposure periods at 700F. Figures 18 through 22 are 20X microphotos that illustrate the change in dispersion of GR 908 in RTV 560 as time at 700F is increased. Additional investigation is suggested in this area.

Flatwise tensile tests were performed on RA 59 and RTV 560 under selected conditions. Specimen configuration is shown in Figure 23 and consists of 2 1/4 inch diameter aluminum blocks bonded together with the test adhesive. Center hole is for a pin which assures alignment of the two halves during bonding. The two specimen halves shown on the right of Figure 23 illustrate the cohesive failure mode (failure within the adhesive rather than between adhesive and load block) which was typical of all specimens tested.

Data shown in Figures 24 and 25 demonstrate a significant advantage in RA 59 at 700F in this specimen configuration. All RA 59 specimen were fabricated using Batch 3 material and RTV 560 specimens utilized Batch 729.

Dynamic weight loss measurements for RA 59, prepared at different weight percentages of curing agent, Thermolyte 12, are shown in Figure 26. A curve

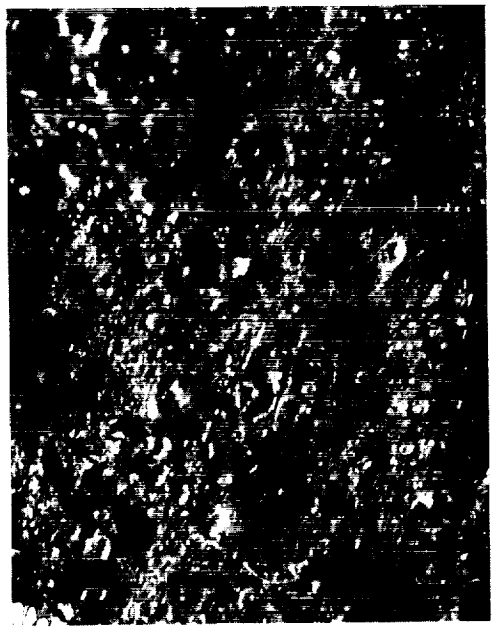


Figure 18. Dispersion - 0.5 Hour at 700F

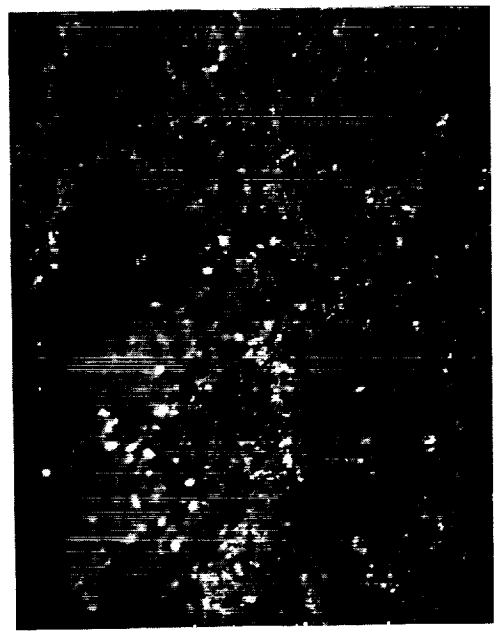


Figure 19. Dispersion - 1 Hour at 700F

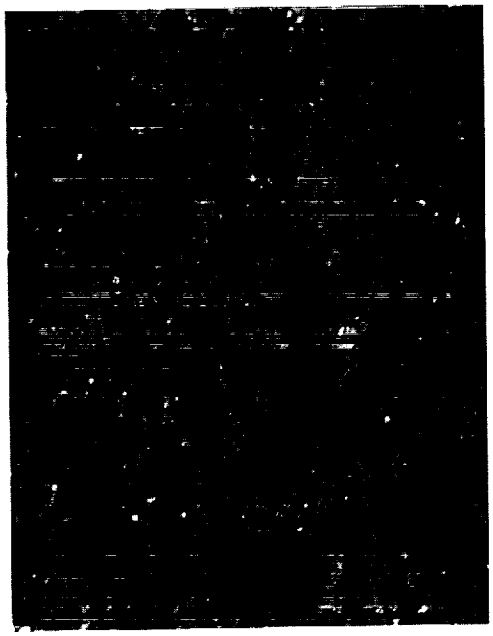


Figure 20. Dispersion - 4 Hours at 700F

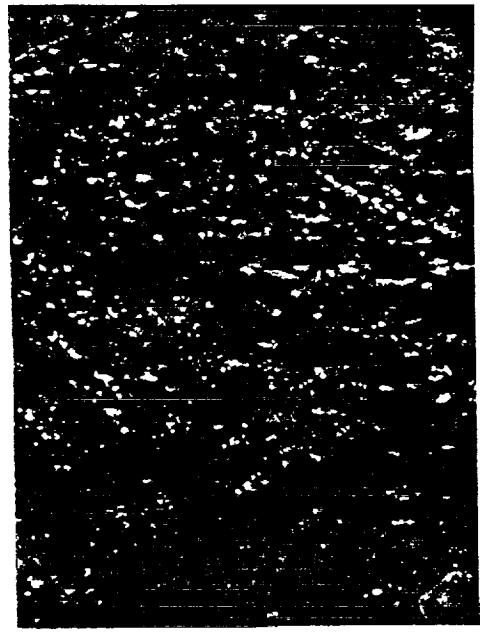


Figure 21. Dispersion - 6 Hours at 700F

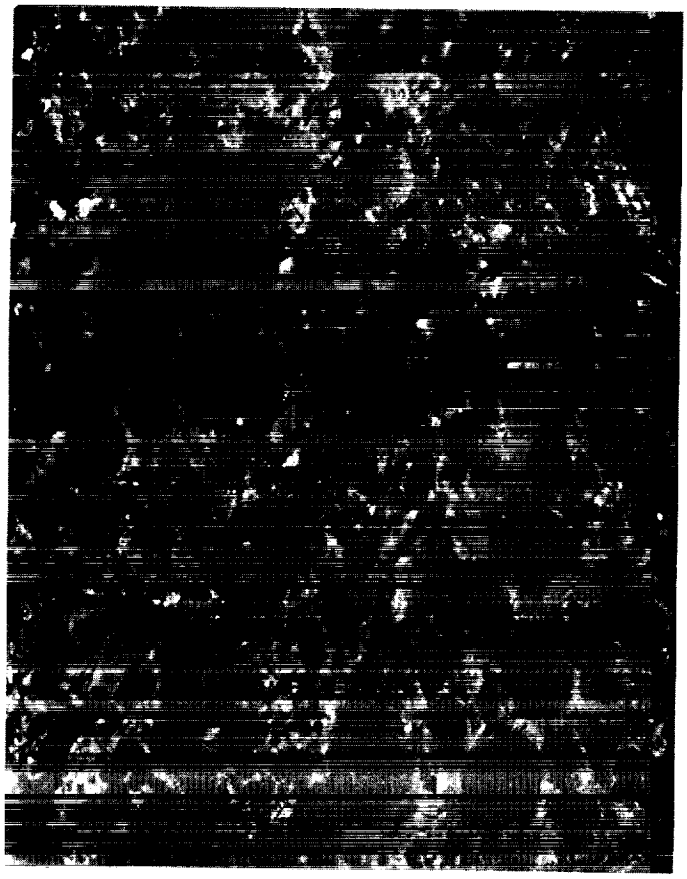


Figure 22. Dispersion - 8 Hours at 700F

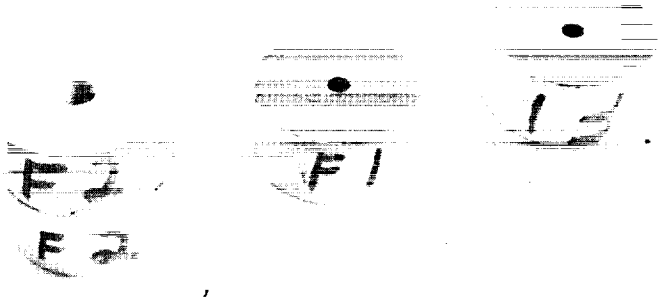


Figure 23. Flatwise Tensile Test Specimens
Before and After Test

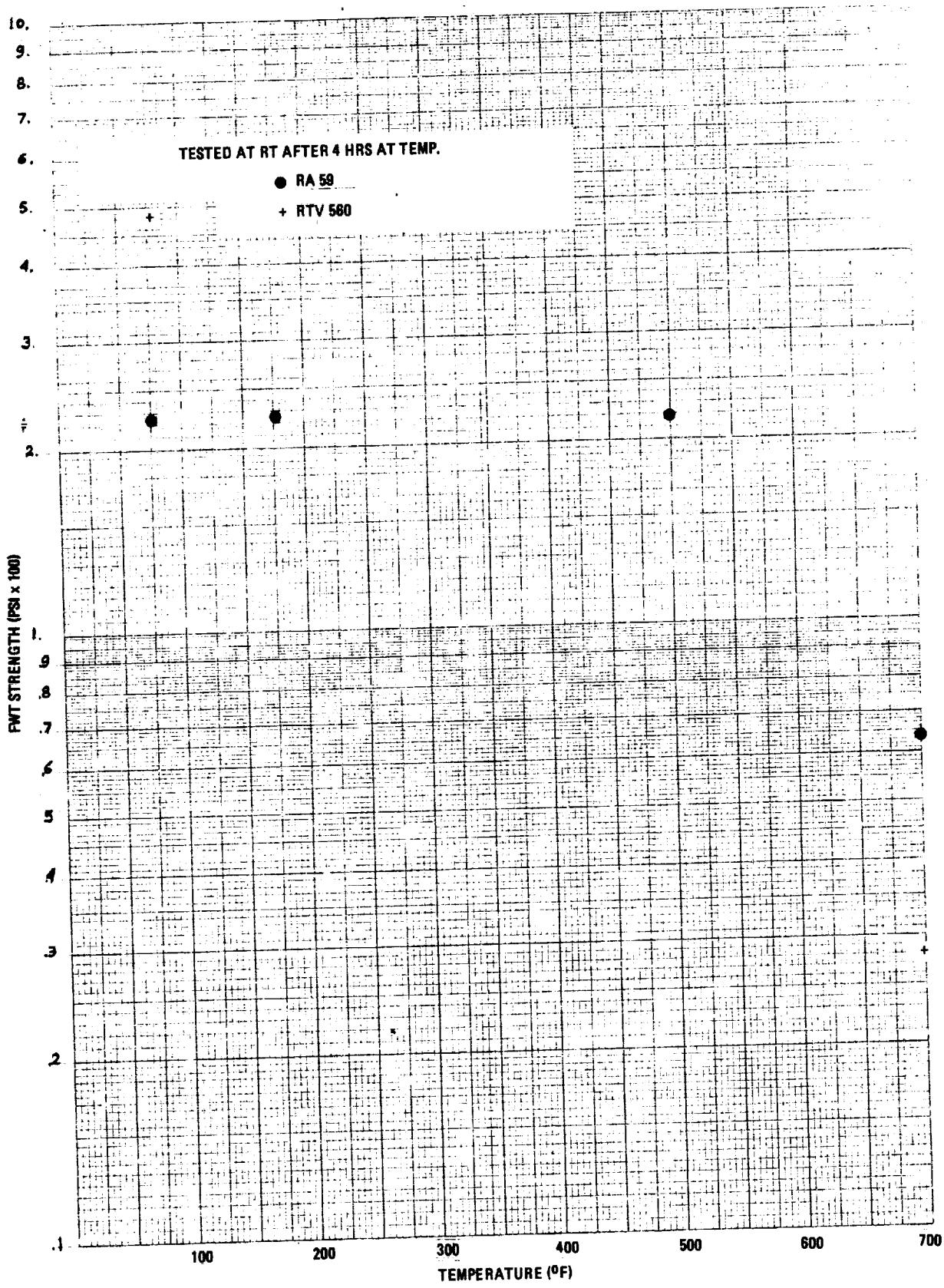


Figure 24. Flatwise Tensile Tests After 4 Hours at Temperature

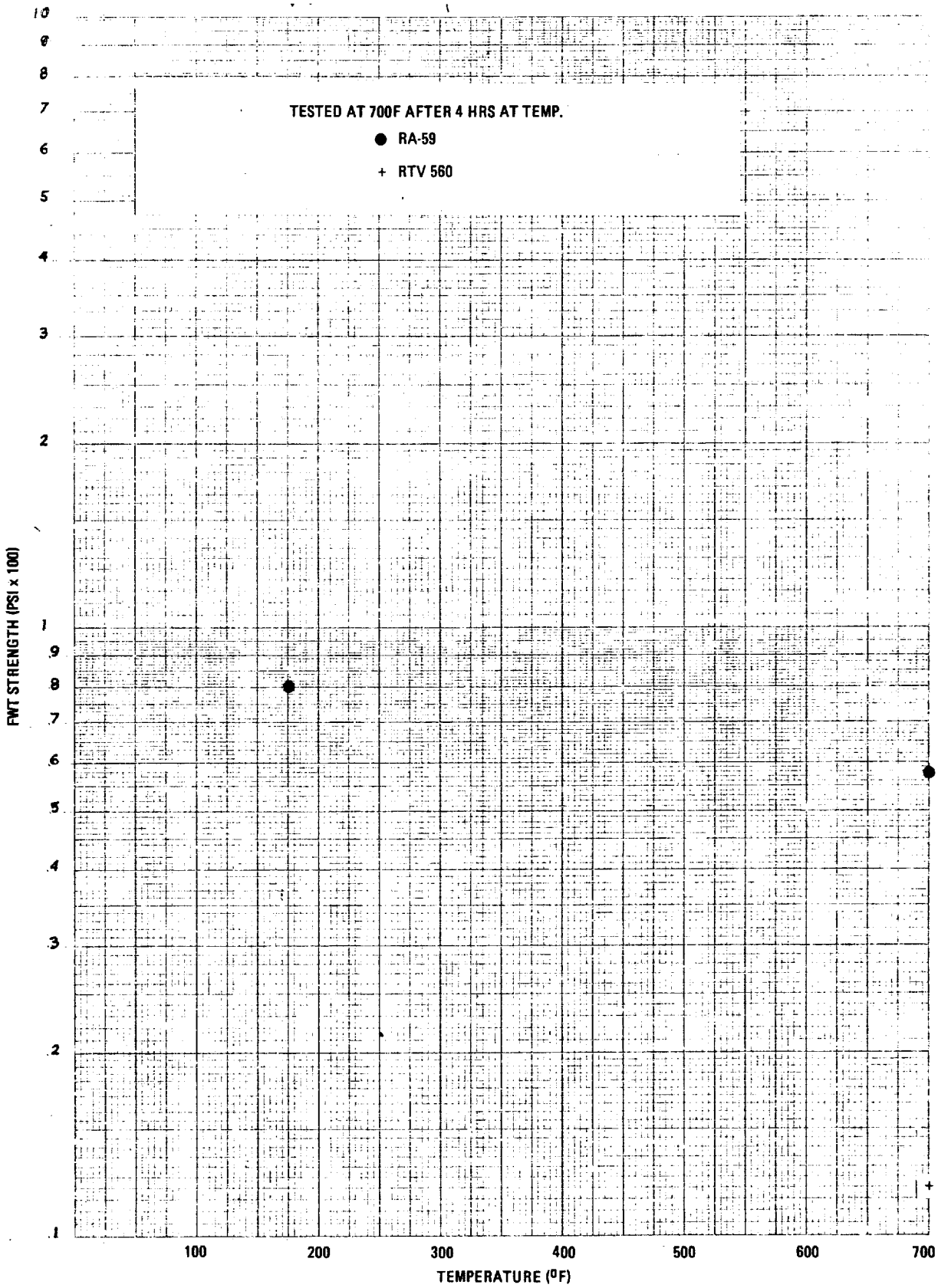


Figure 25. Flatwise Tensile Tests at 700F After 4 Hours at Temperature

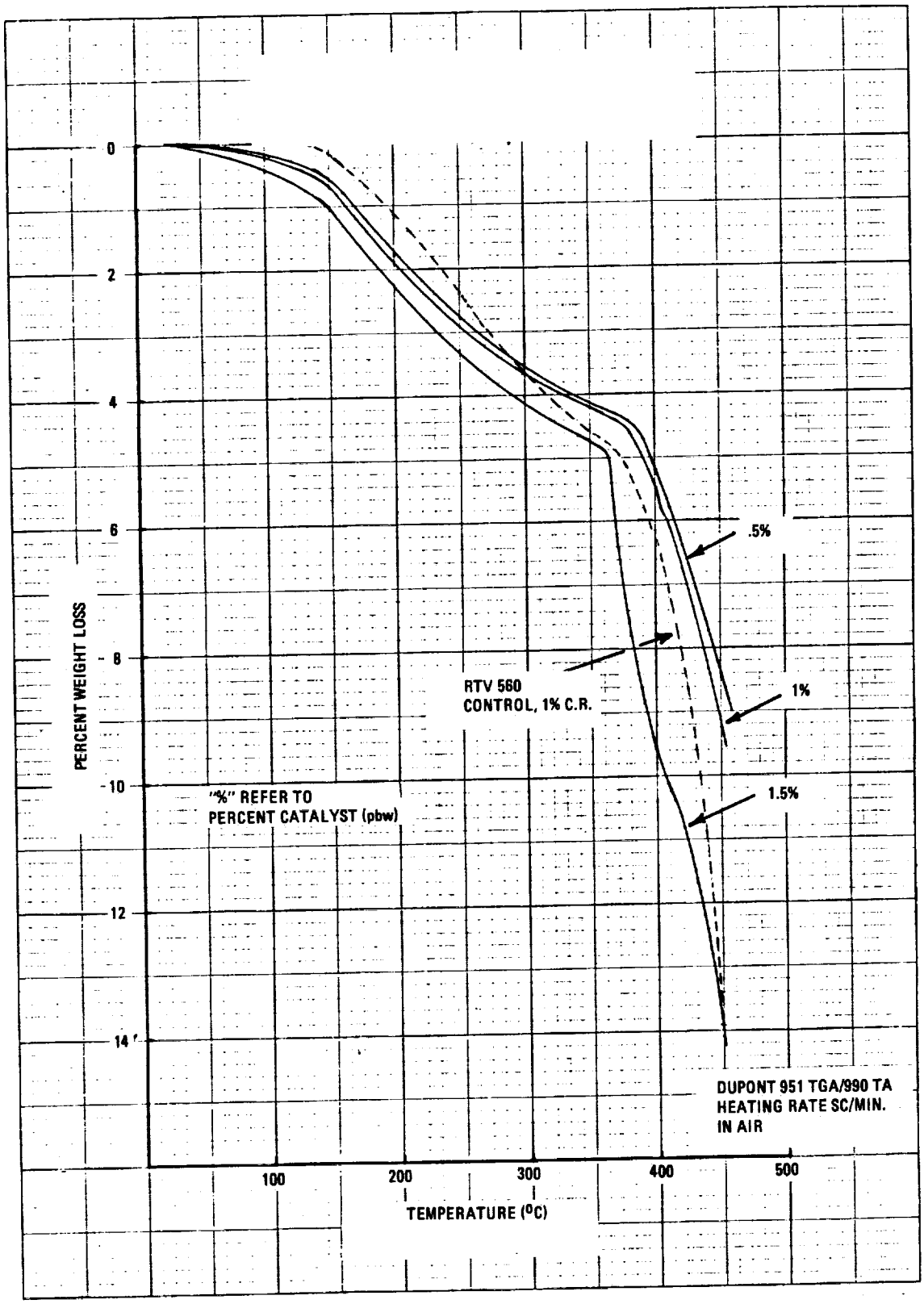


Figure 26. TGA Curves for RA59 Prepared at Different Weight Percentages of Curing Agent (Repeated From Figure 9)

for RTV 560 control material is included for comparison. These curves were obtained with a Dupont 951 TGA module and 990 Thermal Analyzer. The runs were made in air at a heating rate of 5C per minute. The curves show that rapid oxidative degradation begins to occur at 700 to 725F. The differences between RA 59 and RTV 560 are slight with respect to the total weight loss profile to 10 to 15 percent, and the variations obtained would probably be close to batch to batch variations if such a study were made.

Figure 27 presents TMA curves for RA 59 at 1 percent catalyst concentration. The curves were generated with a 10 mil flat tip penetration probe at 20 g, (approximately 50 psi), load. The runs were made with a Dupont 941 TMA and 900 Thermal Analyzer. Heating rate was 5C per minute. The curve for room temperature cured material shows a phase change occurring at 71 to 84C. This phase change is irreversible. The second curve is a re-run of the same sample after exposure to 250C during the first run. The phase change is no longer present. The third curve is for a sample, taken from the same specimen, conditioned at 257C (500F) for 30 to 40 minutes in an air-circulating oven. As can be seen, the phase change is gone.

This phase change is due to the 908 resin present in the RA 59 adhesive. Expansion measurements at zero load with the TMA expansion probe do not show this phase change. Only the RTV 560 glass transition at -118 to -112C is present. The penetration probe at load is required to show this change during expansion. Differential Scanning Calorimetry (DSC) runs on neat 908 resin readily show this phase change. Figure 38 shows an endotherm at 61C for as-received resin. Repeated re-runs on the same sample show the absence of the endotherm but the presence of a slope change characteristic of a glass transition.

PART NO. 941017

DUPONT INSTRUMENTS

SAMPLE: RA 59 1% Catalyst Cast 8/9/78 ORIGIN:	SAMPLE HEIGHT as marked LOADING ON TRAY 20 g PROBE: EXPANSION/PENETRATION HEATING RATE 5 °C/MIN.	X-AXIS SCALE 50 °C/DIV. Y-AXIS SCALE 0.04 MV/IN. Y-AXIS SENSITIVITY 13.2 x 10⁻⁴ IN. PROBE DISPL./IN. OF CHART	RUN NO. DATE 9-8-78 OPERATOR RLM

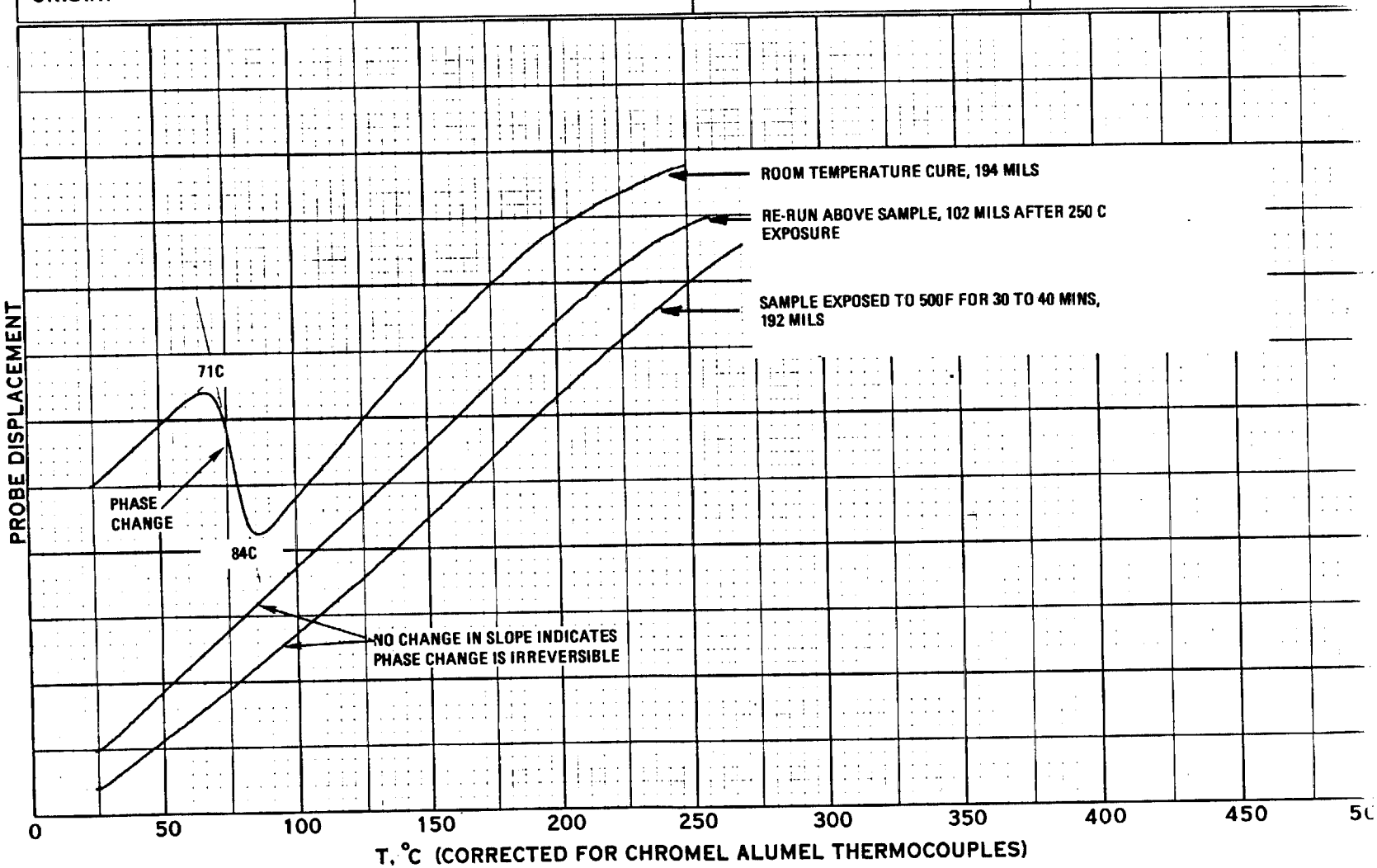
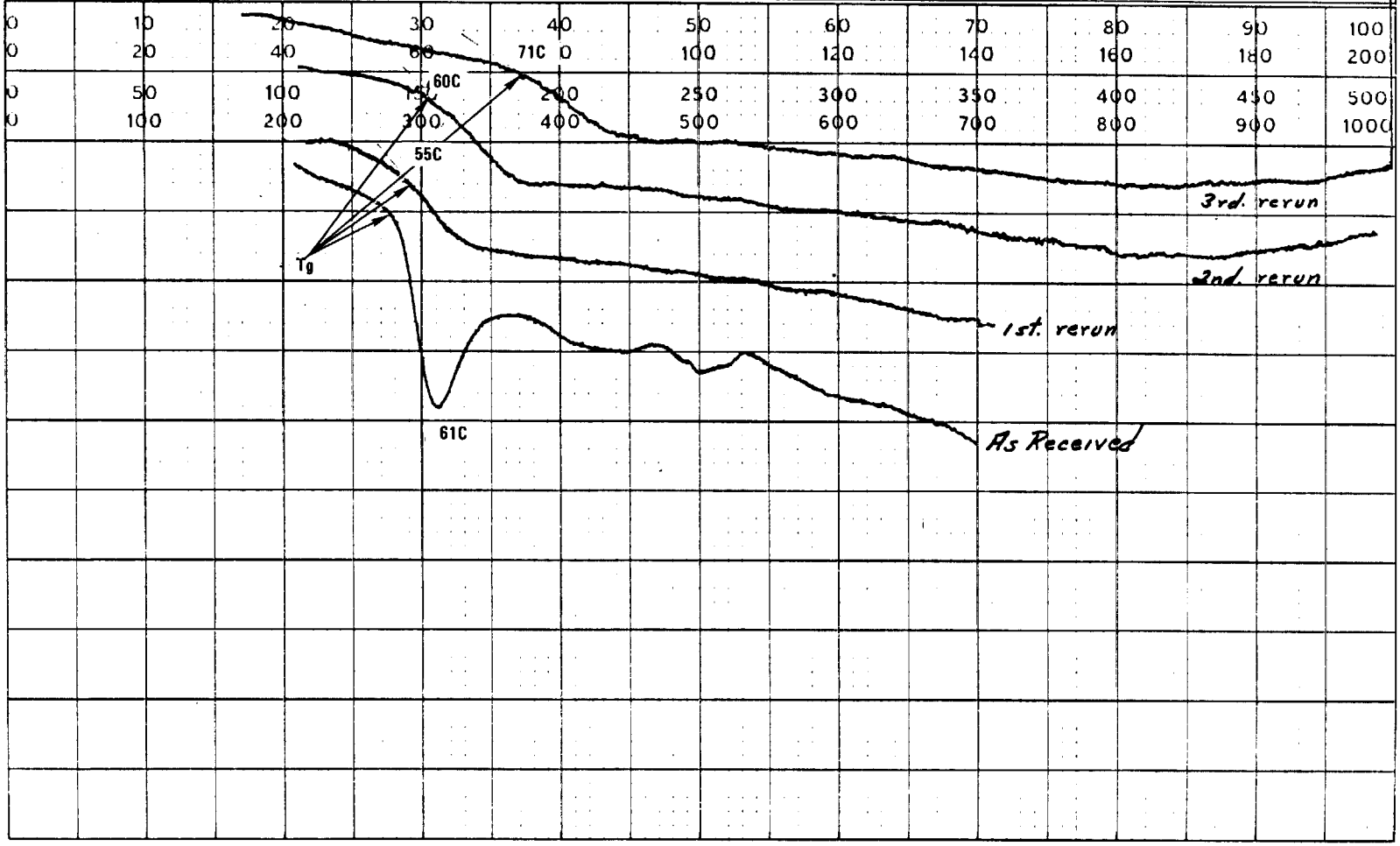


Figure 27. TMA Curves for RA-59 at 1-Percent Catalyst Concentration

PART NO. 900304

DUPONT INSTRUMENTS

SAMPLE: 908 Resin	SIZE —	ATM. Air, 1 atm MM		RUN NO.
	REF. Empty Al Pan		T / △T	DATE 9-20-78
	PROG. MODE Heat	SCALE	20 °C/IN / 0.2 °C/IN	OPERATOR DMM
ORIGIN: Owens-Illinois	RATE 10 °C/MIN, START °C	SHIFT	0 IN. / — IN.	BASE LINE SLOPE 0



T, °C (CHROMEL: ALUMEL)*

* SEE INSTRUCTION MANUAL FOR SCALE CORRECTION

Figure 28. Endotherm of 908 Resin

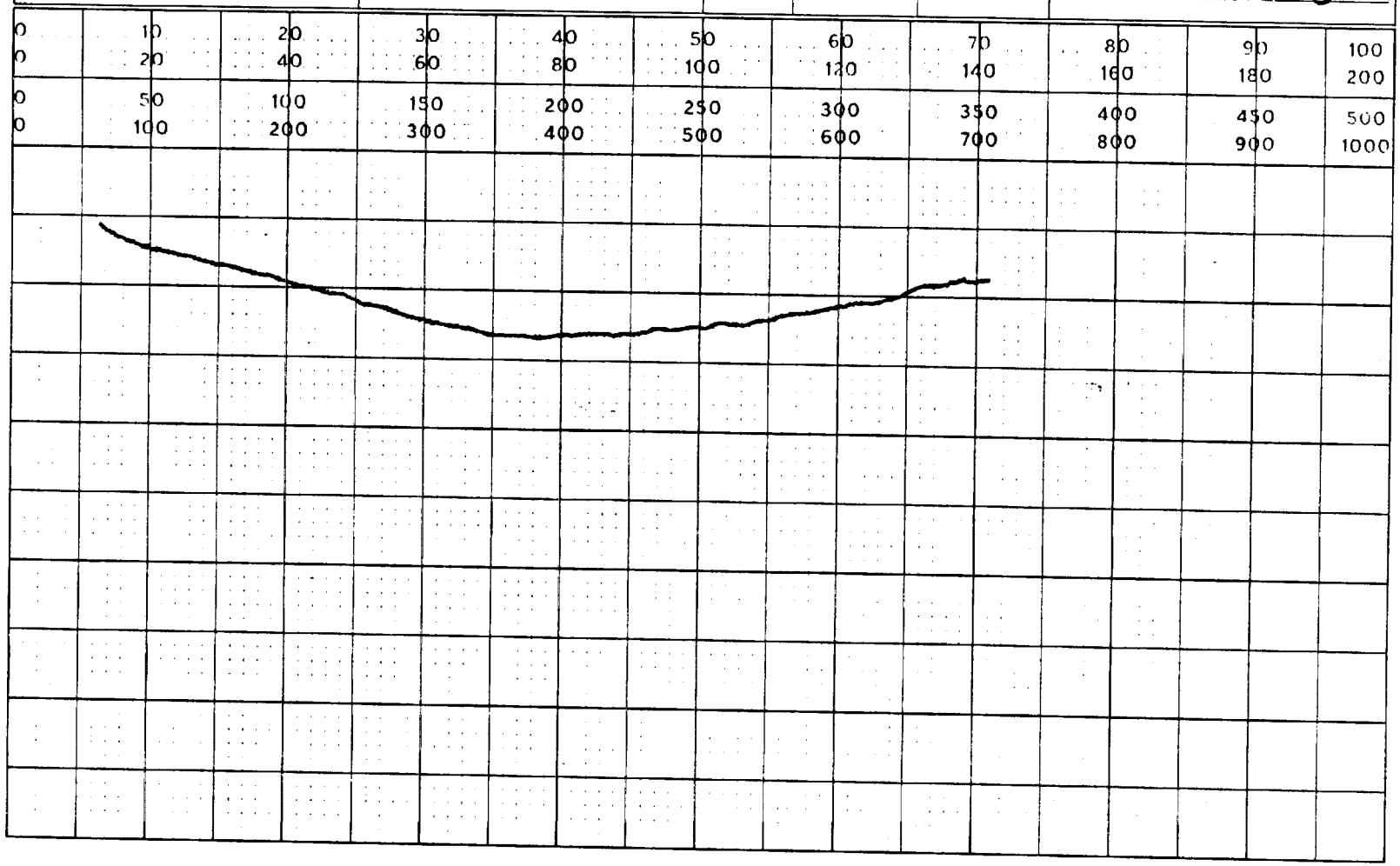
As the re-runs are carried out to higher temperature the temperature of the slope change increases. Figure 29 presents a DSC run on the same sample used in the Figure 28 DSC runs after sample conditioning at 600 F for 1 hour. The apparent glass transition is now gone.

In order to further characterize the 908 resin a film on sodium chloride was prepared for infrared (IR) analyses. The film was cast from carbon tetrachloride, and, after air dry, dried in a vacuum oven for 4 hours at 86F. Figures 30, 31 and 32 show the IR spectra for the same 908 film after, respectively, room temperature application, 300F for 1 hour, and 600F for 1 hour. The spectrum for the 300F conditioning shows a reduction in the absorption bands at approximately 3400 and 890 wavenumbers. After 1 hour at 600F these bands are completely gone. The absorbance at about 3400 can be assigned to the bonded Si-OH stretch and the absorbance at 890 tentatively to the silicone hydroxy assymetric stretch. The spectrum for as-received resin is, generically, typical of a methyl, phenyl silicon.

PART NO. 900304

DUPONT INSTRUMENTS

SAMPLE: 908 Resin Heated for 1 hr in air at 315 C (600 F)	SIZE —	ATM. Air, 1 atm MM	RUN NO.
	REF. Empty Al Pan	T / ΔT	DATE 9-20-78
	PROG. MODE Heat	SCALE 50 °C/IN / 0.2 °C/IN	OPERATOR MM
ORIGIN: Owens-Illinois	RATE 10 °C/MIN, START — °C	SHIFT 0 IN. / — IN.	BASE LINE SLOPE 0



T, °C (CHROMEL: ALUMEL)*

* SEE INSTRUCTION MANUAL FOR SCALE CORRECTION

Figure 29. DSC Run on 908 Resin Sample

Sample 908 Resin
Owens-Illinois
Date 9-20-78
Path – mm
Phase Film on NaCl from CCl4
Comments As-Received

Beckman 4260
Speed 300 cm^{-1}/min.
Gain std.
Period 2
Slit std. mm
Analyst MM

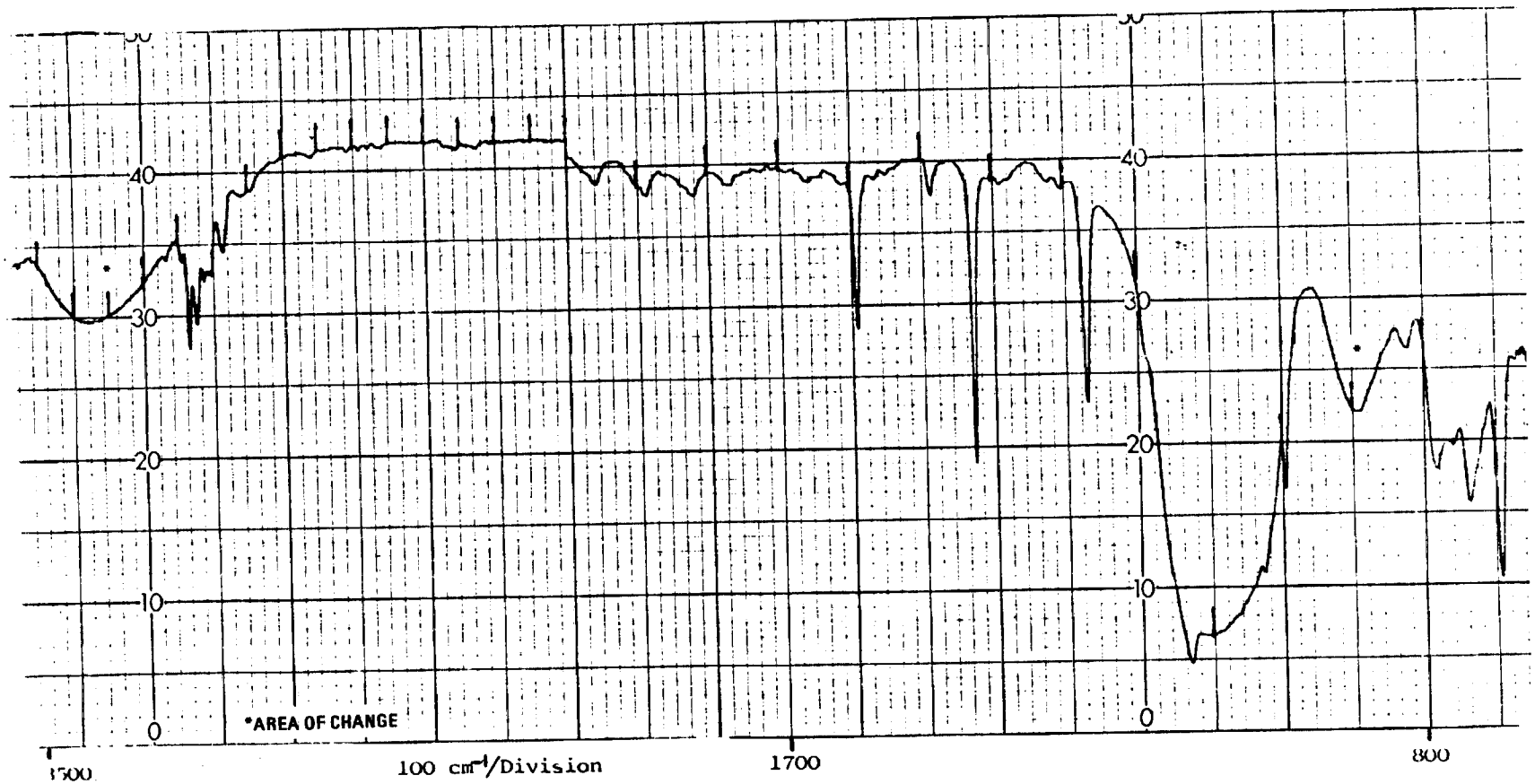


Figure 30. 908 Resin IR Spectra - Room Temperature

Sample 908 Resin

Date 9-20-78

Path – mm

Phase Film on NaCl

Comments Heated 1 hr. @ 300F in air

Beckman 4260

Speed 300 cm^{-1}/min.

Gain std.

Period 2

Slit std. mm

Analyst SMM

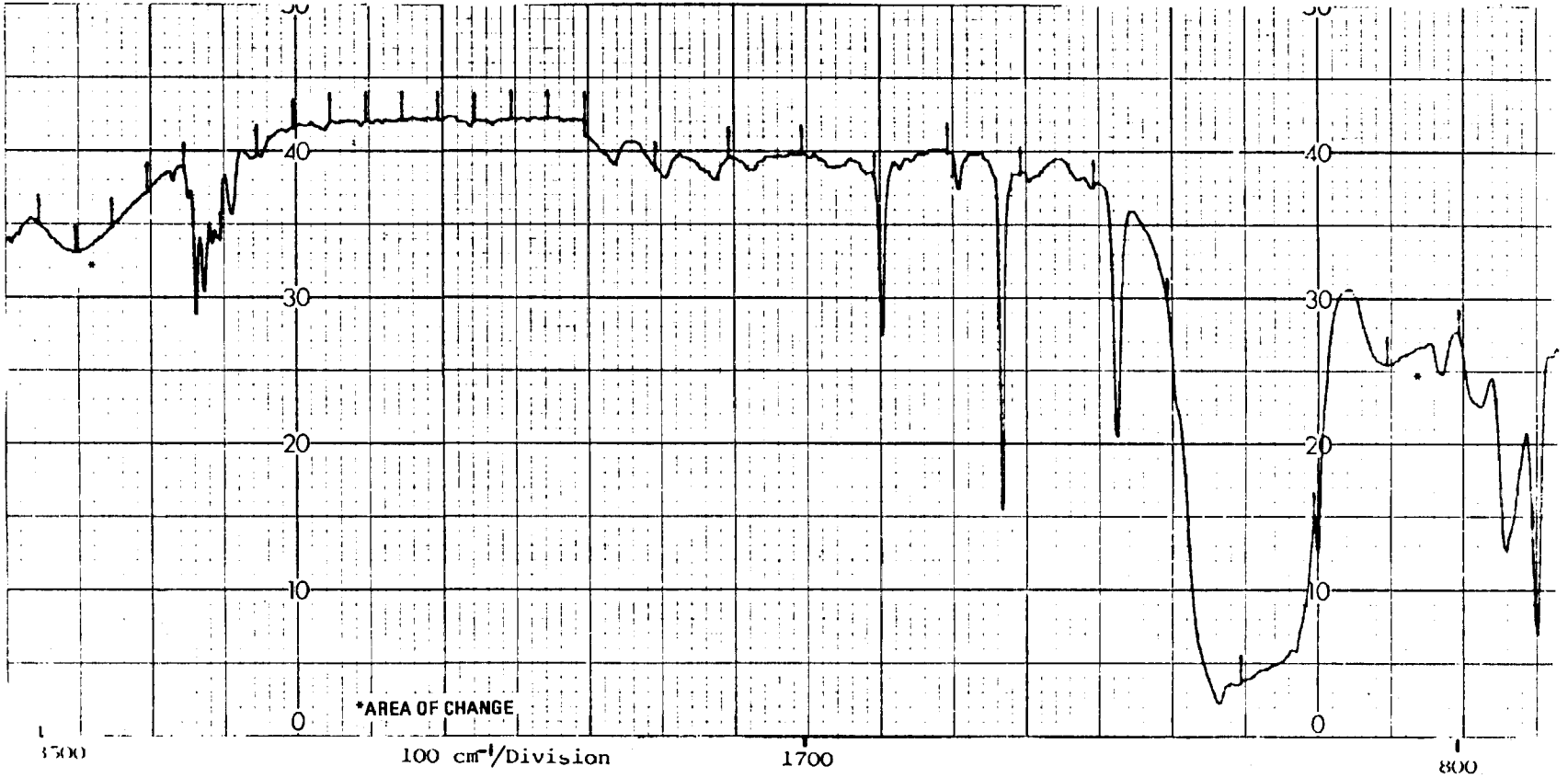


Figure 31. 908 Resin IR Spectra - 300F for 1 Hour

Sample 908 Resin

Date 9-20-78

Path — mm

Phase Film on NaCl

Comments Heated 1hr @ 600F in air

Beckman 4260

Speed 900 cm^{-1}/min.

Gain std.

Period 2

Slit std. mm

Analyst

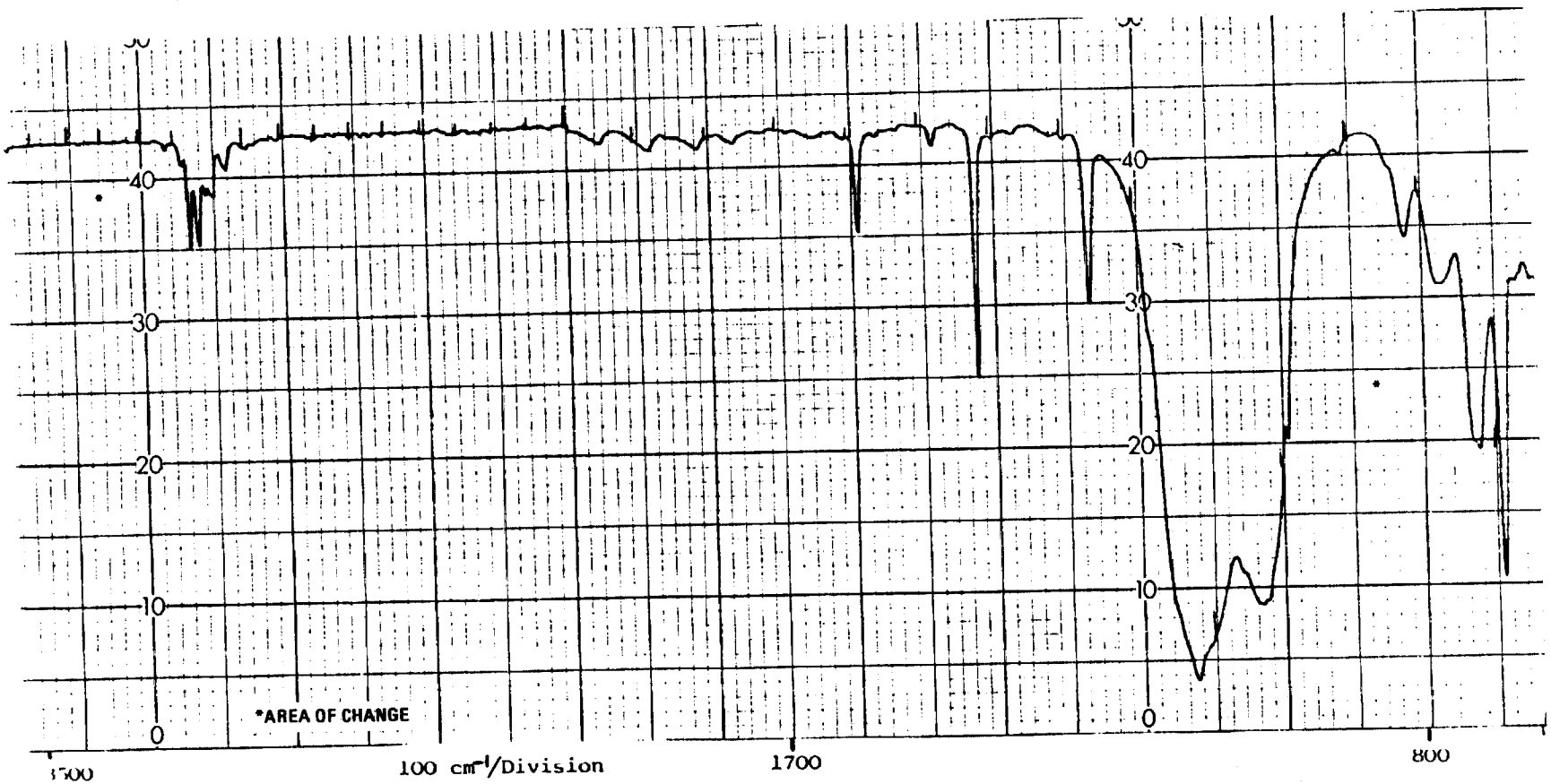


Figure 32. 908 Resin IR Spectra - 600F or 1 Hour

6.4.3 Performance Demonstration

The use of RA 59 as a hard bond adhesive (no SIP) for bonding RSI tiles to polyimide/graphite skin homeycomb sandwich structure was demonstrated. RSI tiles with RCG coating were bonded to the composite honeycomb panel with RA 59 and RTV 560. The panel/tile assembly was cycled several times from room temperature to temperatures down to -300F with a one hour soak at temperature and examined for cracks after each cycle. No cracks were observed after the final cycle. A full tile tensile test was subsequently performed and resulted in tile failure at relatively low loads. Details were as follows:

Components:

Honeycomb Panel - 4 ply skins each 0°, ±45°, 90° skybond 703/eelion 3000 3/16" HRH 4# cure, FM34 adhesive.

Tile = 2-1/2" X 6" X 6", Nos. 0060, 0065, 0075, 0082

RA 59, Batch #2 (Ref. Page -35)

RTV 560, Batch 731

Procedure

Tile bonding surface lightly sanded and blown clean with compressed air.

Adhesive evenly spread over bonding surface as follows:

Tile #0065-RA59, 5 grams

Tile #0060 - RTV560, 4.5 grams

Composite bonding surface scuff sanded and primed with SS4155 (MB0125-050) Batch #LCK 4319. Approx. 0.008" of each adhesive applied to bond surface area. Tile adhesive cured under 4" Hg at R.T. for 24 hours.

Evaluation of adhesive applied to tile bonding surface and exposed to cryogenic temperatures:

#1 - Tile #0022	in., RA 59	Light sand, air blow,
#2 - Tile #0075	in., RTV 560	apply 10 mil (8.3 grams) adhesive to each tile

Specimens described above were located in an environmental chamber and exposed to the following thermal cycles:

Cycle #	Temperature Cycle (°F)	Observations
1	R.T. To -125, 1 Hr. @ -125, -125 to R.T.	No Change
2	R.T. To -175, 1 Hr. @ -175, -175 to R.T.	No Change
3	R.T. To -225, 1 Hr. @ -225, -225 to R.T.	No cracks in bonded tiles. Some shrinkage of RA 59 from one lose tile
4	R.T. To -250, 1 Hr. @ -250, -250 to R.T.	Same as Cycle #3
5	R.T. To -300, 1 Hr. @ -300, -300 to R.T.	Cracks in tile just above adhesive - both tiles, more severe in RA 59. Possible crack in tile at Bond in bonded panel

*No apparent cracks in tile at adhesive interface of bonded specimen. Ice accumulated in gaps between tile corners and substrate, however, no propagation was observed.

Some recession of adhesive from tile edge was noted on both individual specimens. This was more severe on RA 59.

6" X 6" X 1/2" Al load plate bonded to top surface of each tile. Tensile load applied and load at failure recorded.

RTV 560 - 85 lb. load, 36 $in.^2$ area = 2.36 psi, tile failure

RA 59 -160 lb. load, 36 $in.^2$ area = 4.44 psi, tile failure

Figure 33 shows the bonded tile/composite panel prior to thermal cycle and tensile testing. Figures 34 and 35 illustrate tile cracks caused by unrestrained adhesive contraction which occurred during the -300F cycle. Figure 36 shows that failure occurred in the tile rather than at the adhesive bond line during tensile testing. The low contraction coefficient of graphite/polyimide laminate probably restrains normal adhesive contraction for both RTV 560 and RA 59 thus, preventing the type of failure found in adhesive coated but unbonded tiles.

6.5 REFURBISHMENT

It was demonstrated that the RA 59 adhesive could be removed from the GrPi composite substrate by scraping with a dull plastic scraper. This was easily accomplished on specimens regardless of previous thermal conditioning. The substrate could be refurbished by light "scuff" sanding with fine sandpaper.

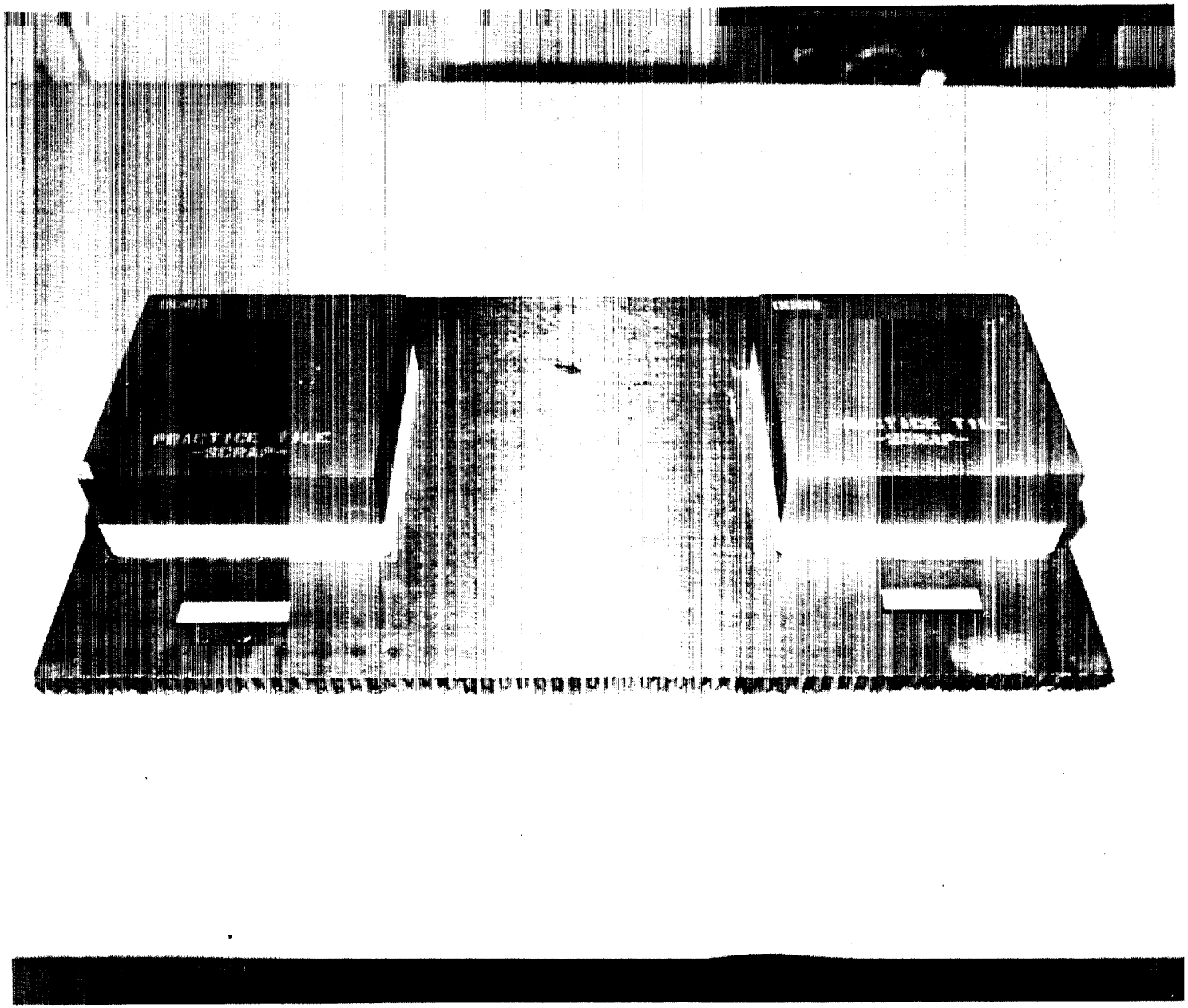


Figure 33. Composite Panel Before Testing

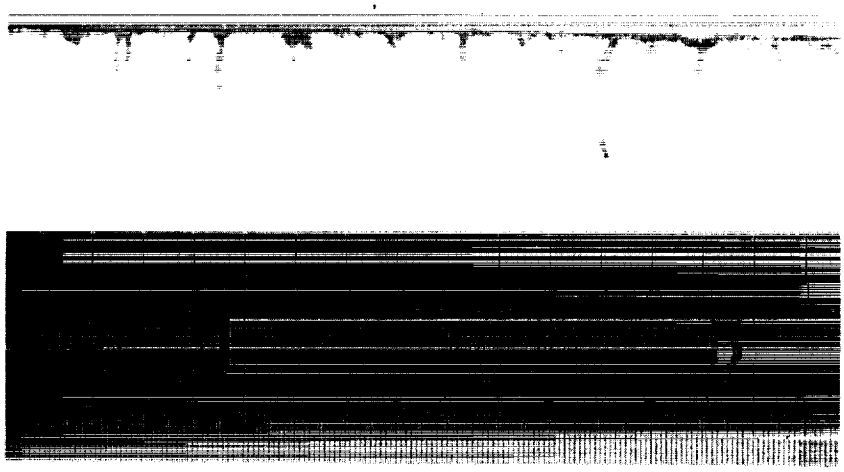


Figure 34. Tile Cracks

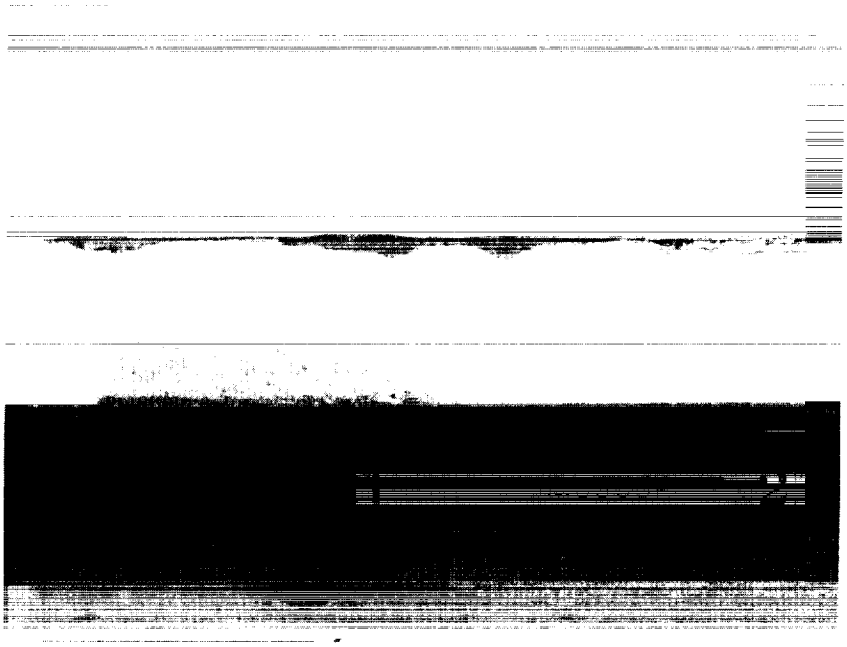


Figure 35. Tile Cracks

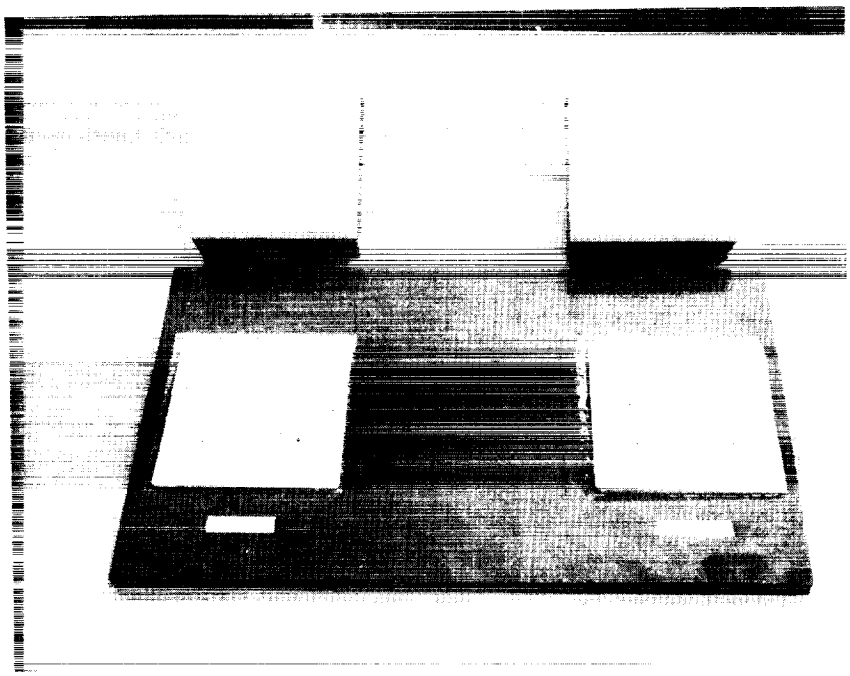


Figure 36. Failed Tile

6.6 Recommendations

Several questions developed during the verification test program which should be considered prior to entering a qualification phase. Specific areas which require further investigation are listed in the following paragraphs.

1. Probably the most important item is determining the curing mechanism (polymerization, cross-linking, etc.) of the two silicone components. The effect of T-12 catalyst on each component, the addition of A-1100 silicone as a catalyst for the OI 908, and the combined effect of these with various amounts of heat on cure mechanism all need study in order to optimize processing and properties.
2. The large data spread which is probably caused by some unidentified variable such as pot life should be determined. This should lead to the ability to obtain consistently high values at all temperature ranges and thus, would show a clear advantage for RA-59 over RTV560. for high-temperature applications.

3. Direct bonding thermal cycle and tensile tests should be conducted with 22PCF tile projected for use on orbiter. Previous tests with L1900 tile resulted in all failures being within the tile at low tensile values characteristic of the latter. The use of higher strength tile should give a better indication of the actual strength of the adhesive or bond.
4. The phenomenon of GR 908 dispersion within the RTV 560 matrix with heat addition should be investigated. There is some indication this is a function of RTV 560 batch variation; thus, the effect of iron oxide particle size within RTV 560 would be included in the study.
5. A study of the cure mechanism of GR 908 alone was performed using IR techniques; however, this could not be accomplished with the RA 59 mixture due to iron oxide within the RTV 560. An attempt should be made to obtain "unfilled" RTV 560 so that this technique can be extended to the mixed materials.
6. Quality control of both GR 908 and RTV 560 should be investigated at the source to identify critical parameters and determine the effects of variability in both raw material quality and subsequent performance of RA 59.

APPENDIX A

EXPERIMENTAL RESULTS

11 NOV 1977 TO 1 MARCH 1978

PREPARED BY: C. L. HAMERMESH

INTERIM SUMMARY REPORT

ADHESIVE BONDING OF RSI TILES TO GRAPHITE/ POLYIMIDE COMPOSITE

Summary:

RTV560 mastic has been successfully modified to extend the high temperature working range without affecting the method of mastic application or the room temperature cure properties. Lap shear stress tests show a twofold increase in strength at 685°F. Flatwise tensile tests show a fourfold increase in bond strength at 700°F. Room temperature and 600°F measurements are either unchanged or enhanced. This improvement has been obtained by adding 30% by weight of Owens-Illinois Type 650 glass resin (a sesquisiloxane polymer) as an acetone solution to the RTV560 mastic, evaporating off the excess solvent at room temperature, and subsequently curing at room temperature with the addition of dibutyltin dilaurate catalyst in the same manner as is done with the RTV560 mastic alone.

Discussion:

Sesquisiloxanes are heat-setting silicone polymers. Owens-Illinois Type 650 glass resin is so called because the flake form has a glassy appearance. It is easily pulverized to a fine powder with a mortar and pestle, however. The initial approach, which was an attempt to prepare a room temperature curing mastic from the glass resin alone, was not successful.

The next approach was to develop an RTV-glass resin mastic mixture. Compatible mixtures were obtained in the range 0-45.8% by weight of glass resin in RTV560 mastic. The Type 650 glass resin was first dissolved in acetone, then the acetone solution was slowly added with stirring to the RTV560 mastic. The excess solvent was allowed to evaporate off in a hood at room temperature, with frequent stirring (every 1/2 hour) to prevent lumping of the glass resin. A 61% mixture of glass resin in mastic was prepared, but difficulty was encountered in obtaining a uniform mixture without gellation of the glass resin, and the high temperature lap shears were, in fact, lower than with lesser concentrations of glass resin.

Preliminary results with the Type 650 glass resin indicate that mixing glass resin with RTV560 results in a mastic with the desired properties of satisfactory bond strength to 700°F, room temperature cure, spreadable consistency, low VCM, and no toxicity.

Lap shear stress tests were run at room temperature, 600°F, and 685°F with a 30.5% glass resin - 69.5% RTV560 mixture. RTV560 samples were run as controls. In all cases the bond strengths were at least as good as those of the controls, and were superior at the high temperature (685°) (Fig. A-1, Table A-1).

TABLE A-1

LAPSHEAR STRESS VALUES, PSI

TEMP, ^{o}F	30.5% GR	RTV-560
22^{o}	54.8	51.4
600^{o}	20.7	20.1
685	24.0	10.8

Next, experiments were performed to optimize the composition of the mastic mixture. At 61% glass resin, poor results were obtained. Separation of the glass resin was found to be a problem. Mixtures of 22.9% and 45.8% glass resin gave good results at room temperature and 600°F, but were poorer at 685°F (Table A-2, Figs. A-2 through A-5). Room temperature lap shear stress measurements made after excursion to 600°F for 10 minutes clearly show the superior potential of the 30.5% glass resin composition (Fig. A-6, Table A-3). These results show promise for this mixture to perform well in cycling tests to be carried out in the next phase of the work effort.

In order to test the glass-resin mastic mixture in a manner more closely approximating the use situation, flatwise tensile tests were performed on the 30.5% glass resin mixture. A layer of SIP felt was sandwiched between two graphite-polyimide composite pieces, using the mastic mixture on each piece of graphite. Again RTV560 served as the control. Results at room temperature were essentially the same for the control and the test mixture. At 600°F, the new mixture was twice as strong, and at 700°F the control mastic experienced definite cohesive failure at the GR/PI composite - SIP interface, while the glass resin mixture did not fail. Only SIP stretching was seen, even after 15 minutes of such stretching. Failure of the adhesive could not be obtained (Table A-4). To get some measure of relative bond strengths, the flatwise tensile tests were performed at

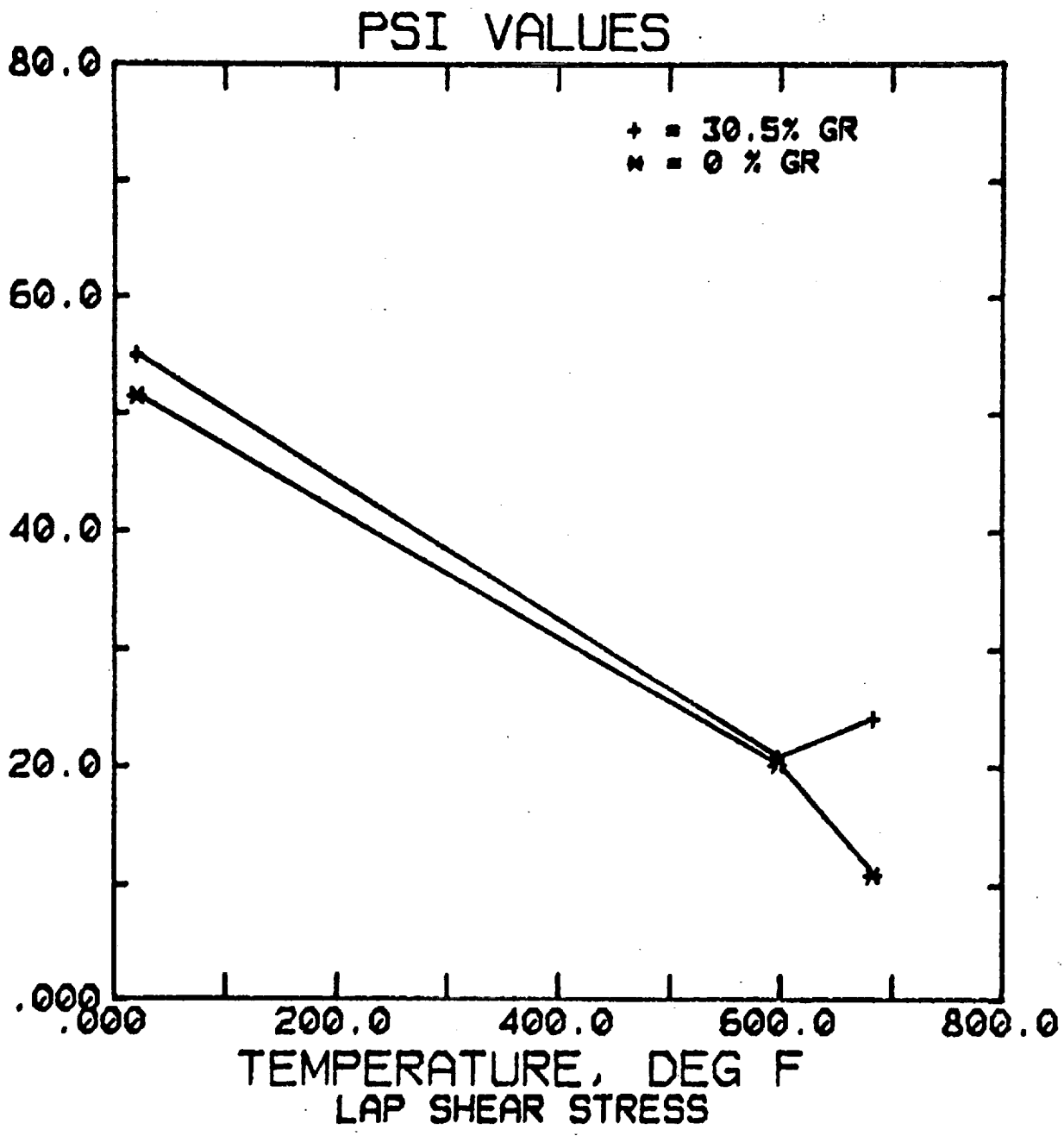

PSI VALUES
+ = 30.5% GR
* = 0 % GR
80.0
60.0
40.0
20.0
.000
.000
200.0
400.0
600.0
800.0
TEMPERATURE, DEG F
LAP SHEAR STRESS

Figure A-1

TABLE A-2

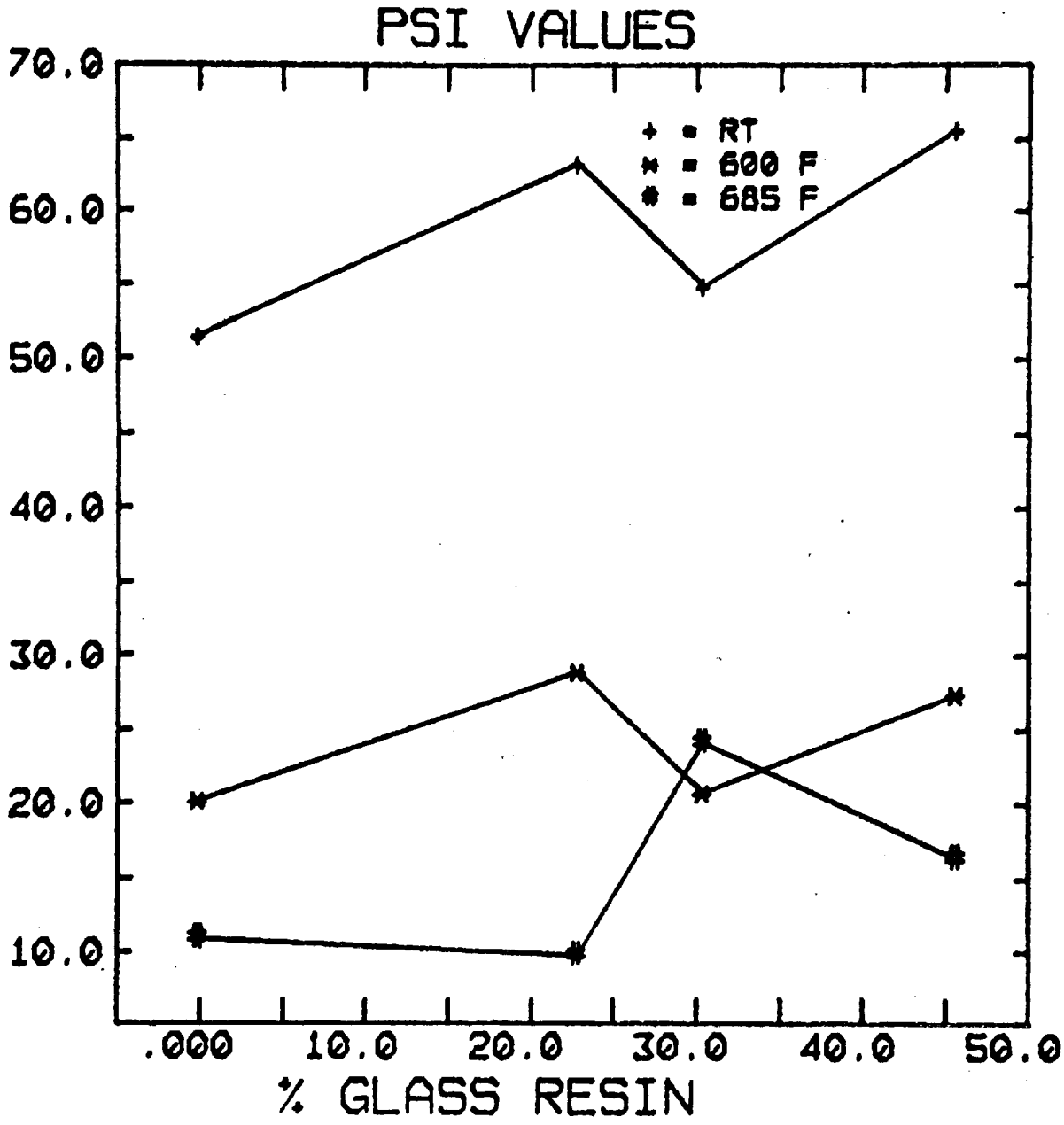
LAP SHEARS (PSI) FOR RTV560-GLASS RESIN MASTICS

	% GR	RT	600	685
1:	.000	33.2	20.1	10.8
2:	22.9	32.4	28.8	9.70
3:	30.5	102.	20.7	24.0
4:	45.8	20.5	27.3	16.3

TABLE A-3

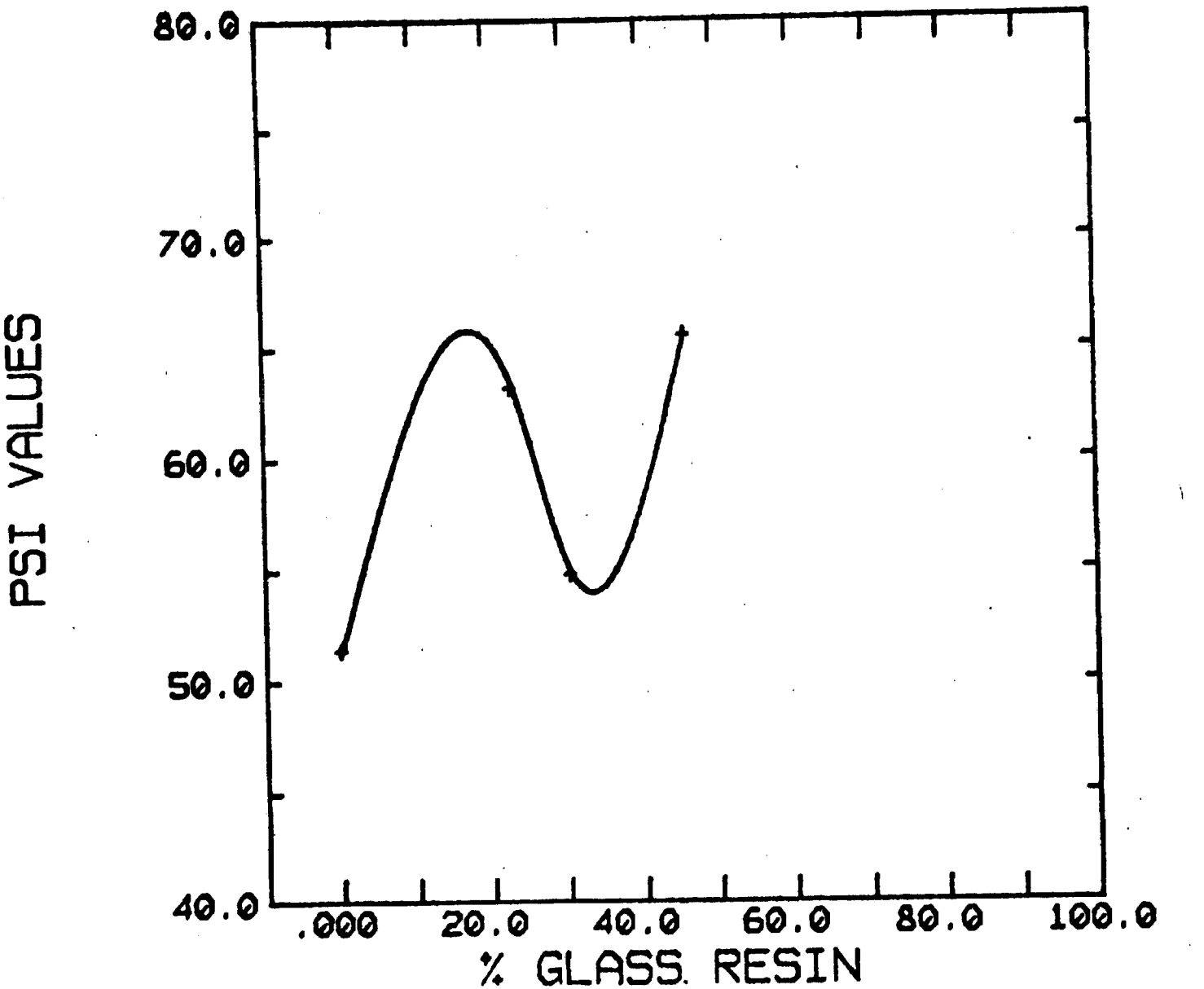
LAP SHEARS AT ROOM TEMP (EXCURSION)

	% GR	PSI
1:	.000	33.2
2:	22.9	32.4
3:	30.5	102.
4:	45.8	20.5



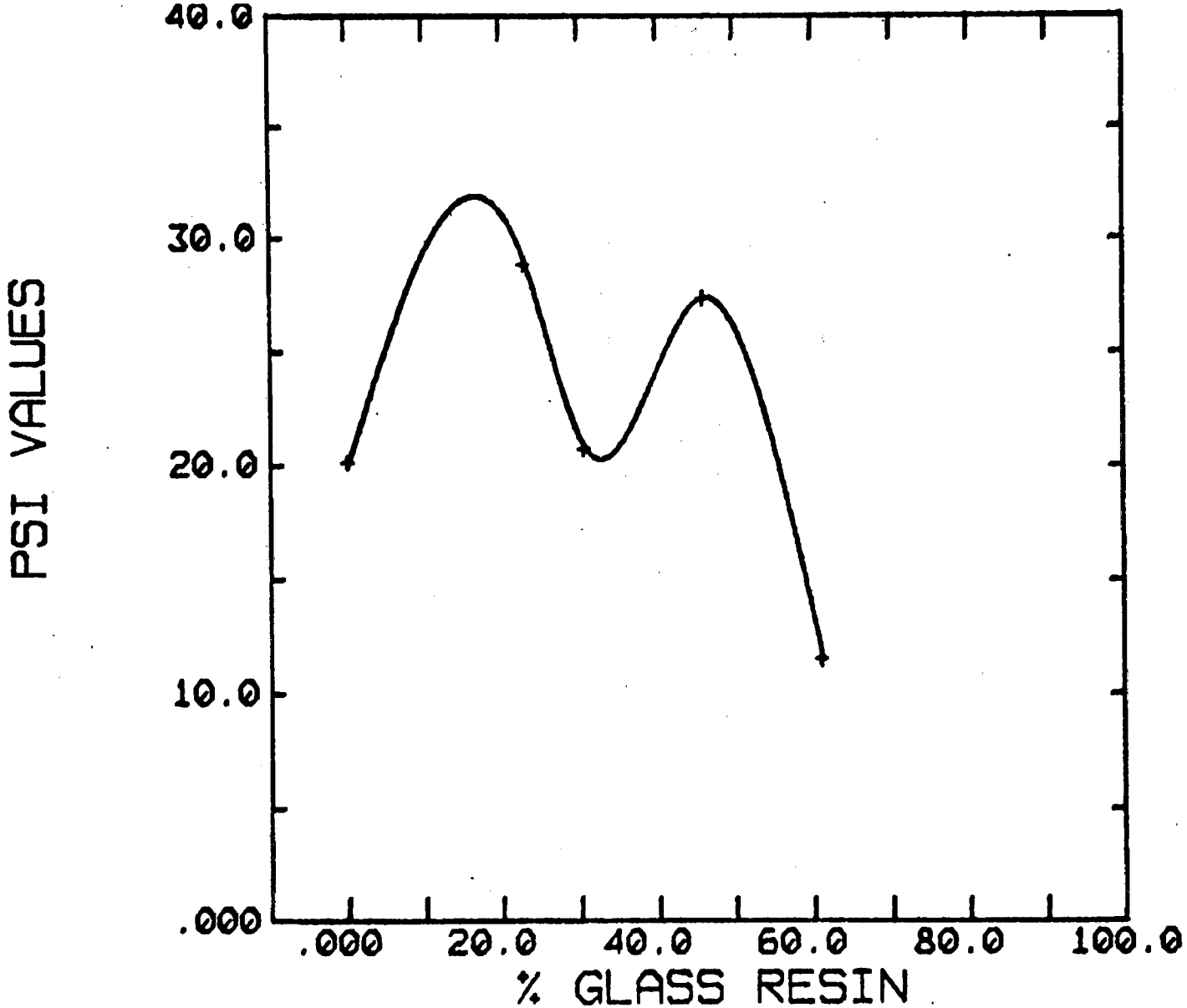
LAP SHEAR STRESS FOR 560RTV-GLASS RESIN MASTICS

Figure A-2



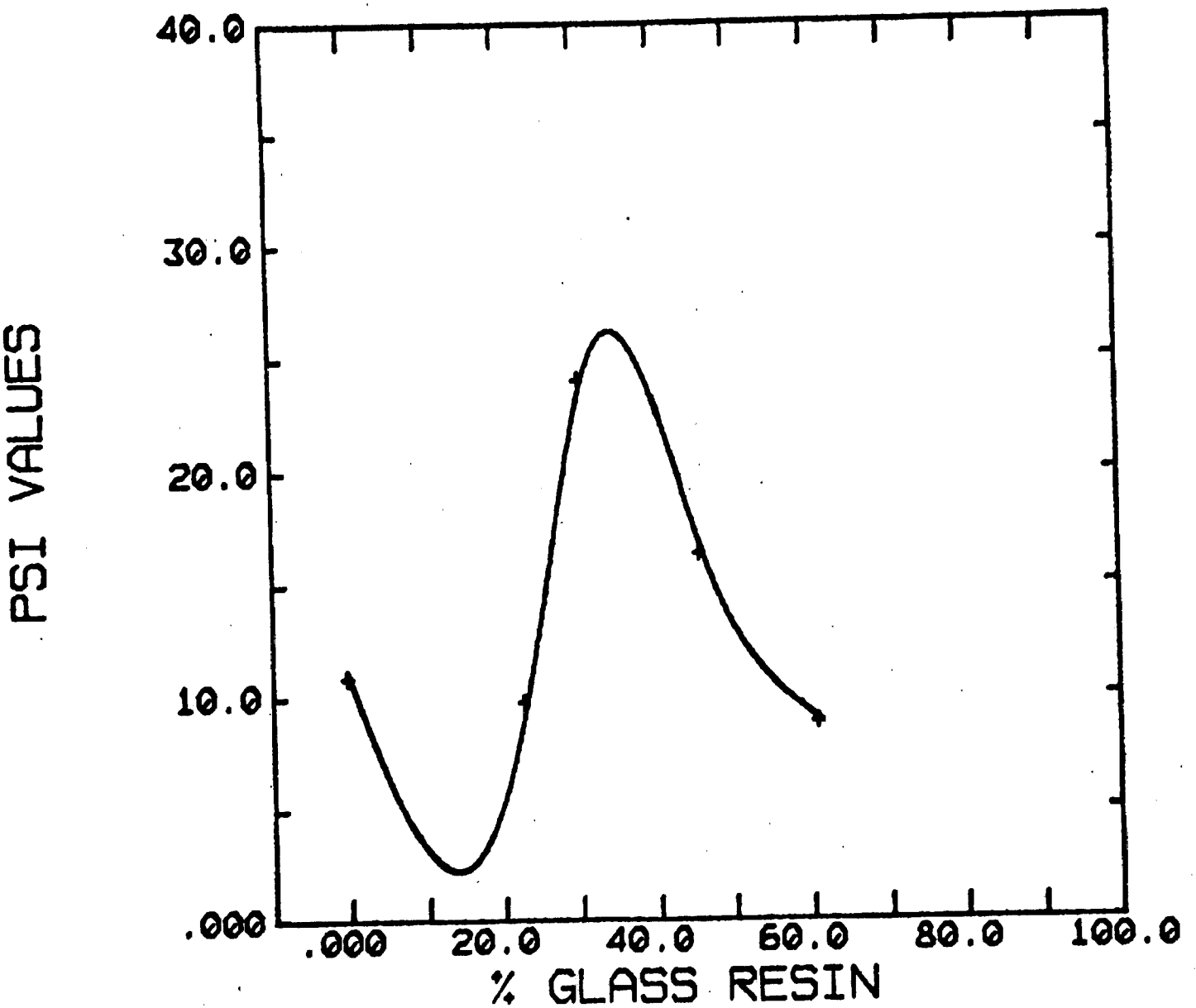
ROOM TEMP LAP SHEAR STRESS FOR 560-GLASS RESIN MASTICS

Figure A-3



LAP SHEAR STRESS FOR 560-GLASS RESIN MASTICS AT 600 F

Figure A-4



LAP SHEAR STRESS FOR 560-GLASS RESIN MASTICS AT 685 F

Figure A-5

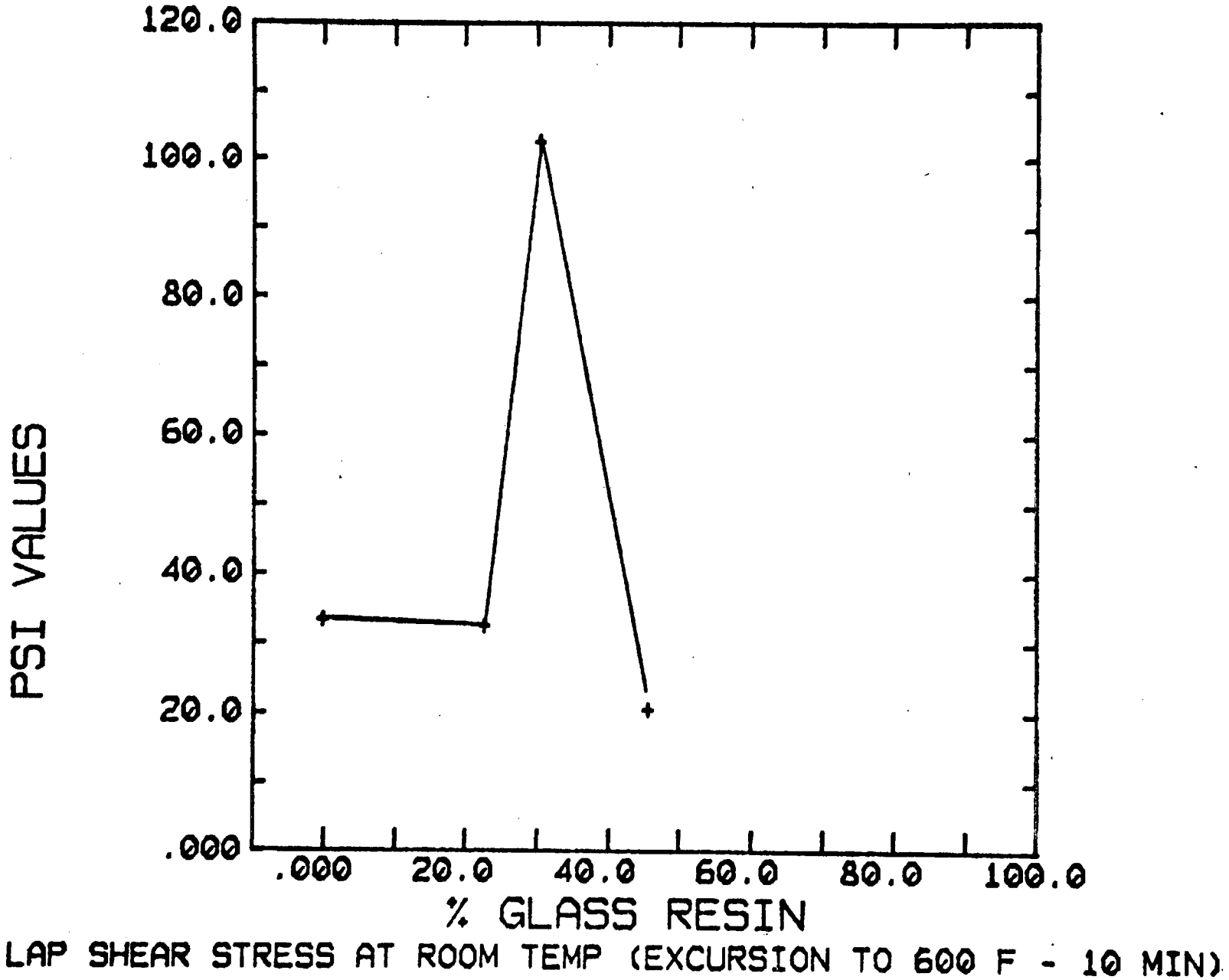

120.0
100.0
80.0
60.0
40.0
20.0
.000
PSI VALUES
.000
20.0
40.0
60.0
80.0
100.0
% GLASS RESIN
LAP SHEAR STRESS AT ROOM TEMP (EXCURSION TO 600 F - 10 MIN)

Figure A-6

TABLE A-4

FLATWISE TENSILE STRENGTHS OF GLASS RESIN - RTV MASTICS

WITH SIP

	RTV 560		GLASS RESIN - RTV MASTIC	
	PSI	FAILURE MODE	PSI	FAILURE MODE
AMBIENT	17.4	INTERFACIAL	19.4	INTERFACIAL
600°F	7.9	INTERFACIAL	18.1	INTERFACIAL
700°F	23.9	COHESIVE	26.1	SIP STRETCHING * (NO FAILURE OF MASTIC)

* TENSILE STRENGTH OF SIP = 20-40 PSI

WITHOUT SIP

	RTV 560	GLASS RESIN-RTV MASTIC
700°F	1.2 PSI	4.8 PSI

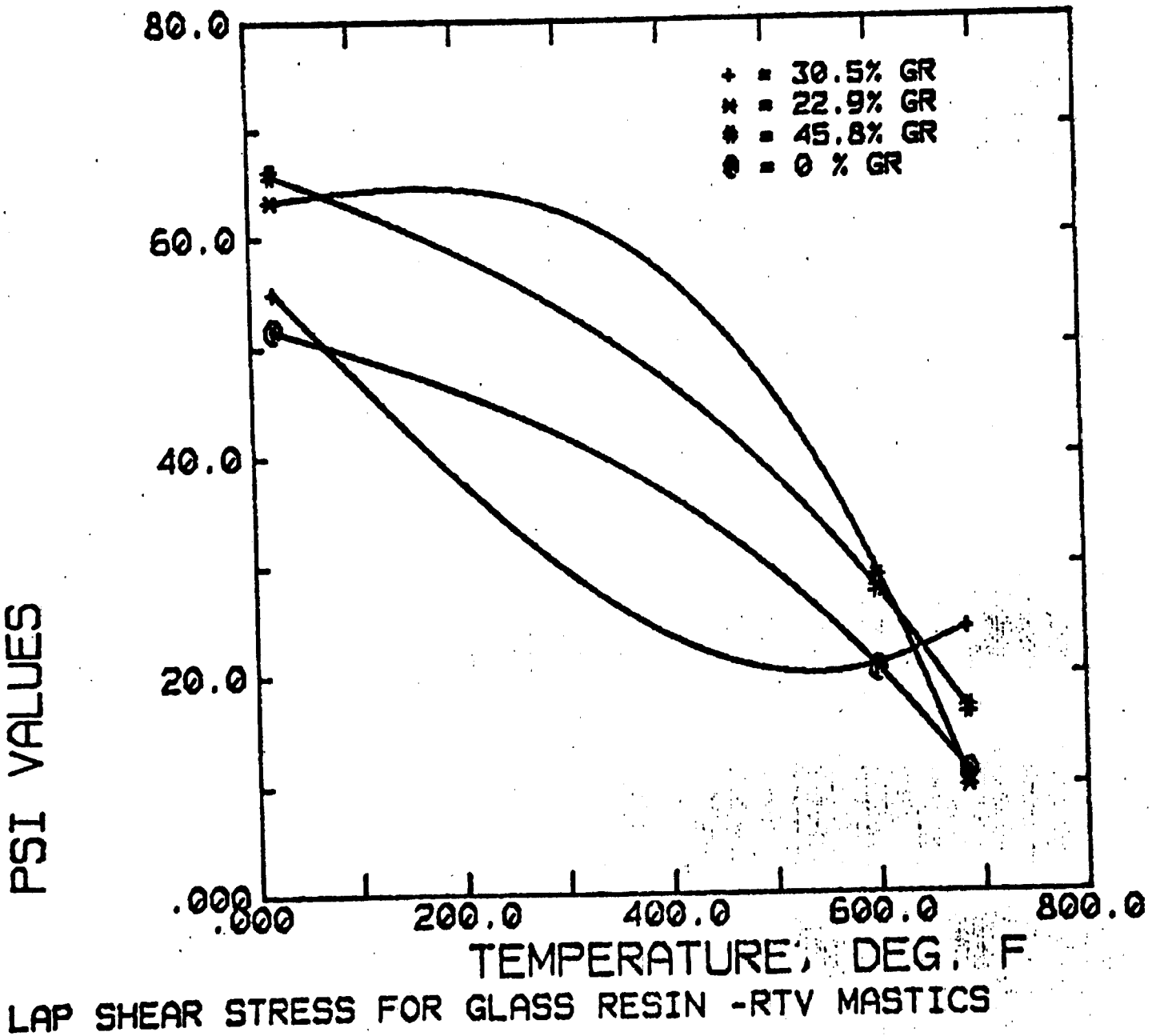
700°F without the SIP layer. The glass resin - RTV560 mastic was four times stronger than RTV560 alone (Table A-4).

Experiments were also carried out to determine the influence of a primer on the bond strength. As expected, bond strengths were increased at room temperature. At 685°F, however, the primer had virtually no effect on the measurements obtained (Table A-5).

The individual behavior of the various mixtures of glass resin - mastic at the test temperatures is shown in Table A-6 and Fig. A-7.

Experimental Details:

Lap Shear Stress Tests - Lap shear samples were prepared using strips of graphite/polyimide composite (GR/PI) which were 1" wide and 4" long. Before bonding, the strips were sandblasted, dusted, and then washed with methylene chloride and allowed to air dry. An overlap of 1/2" formed a bond area of 1" x 1/2" or 1/2 in^2. After the catalyst was added to the mastic, it was thoroughly mixed, and spread after waiting a minimum of 10 minutes. The mastic was carefully spread into each bonding surface, and then a thin layer of mastic was smoothed on top of both surfaces before the overlap was made. Shims of GR/PI composite were placed under the samples to keep the bond line even. Weights were placed on the samples during



LAP SHEAR STRESS FOR GLASS RESIN -RTV MASTICS

Figure A-7

TABLE A-5

EFFECT OF PRIMER ON LAP SHEAR STRESS VALUES

	RTV 560		RTV 560-GLASS RESIN (30.5%)	
	Primed	Unprimed	Primed	Unprimed
R.T.	148.7	51.4	170.3	54.8
685°F	11.9	10.8	24.9	24.0

TABLE A-6

LAP SHEAR STRESS FOR 560RTV-GLASS RESIN MASTICS

	TEMP	0 % GR	22.9%GR	30.5%GR	45.8%GR	61% GR
1:	22.0	51.4	63.2	54.8	65.6	-
2:	600	20.1	28.8	20.7	27.3	11.5
3.	685	10.8	9.70	24.0	16.3	8.80

the cure period (usually overnight or longer). The measurements were obtained on an INSTRON tester, using a crosshead speed of 0.01 cm/min.

Primer Tests - After sandblasting and washing with methylene chloride, the bonding ends of the GR/PI strips were wiped lightly with an adhesive primer, GE 554155 silicone primer, which is a cobalt-blue-colored thin liquid. This was allowed to air dry and then mastic was applied as above.

Flatwise Tensile Tests - Brass cylinders were prepared for bonding by first wiping the surface with methylene chloride, allowing it to air dry, and then dipping in a $FeCl_3$-HCl bath for 1-2 minutes. The bath consists of 50 parts concentrated HCl, 20 parts $FeCl_3 \cdot 6H_2O$, and 30 parts water. The cylinders were rinsed with distilled water and air dried. Then a 1"x1" square of film adhesive HT424 was placed on the surface, and a dry 1"x1'1/4" piece of graphite/polyimide composite, which had been previously sandblasted on both sides and washed in methylene chloride, was placed on top. These were baked at 350°F for 1 hour with weights for pressure, then cooled to room temperature with weights still applied. The exposed surface of the brass cylinder was wiped with a silicone mold release. A 1"x1" piece of SIP was cut for each sample. Mastic was rubbed in well

on the GR/PI surface, then a film of mastic was spread on, and the SIP piece was sandwiched between two such specimens. The samples were held together with weights applied, and allowed to cure at least 24 hours before testing.

Conclusions:

The addition of sesquisiloxane polymer to RTV560 mastic imparts high temperature stability to the mastic and increases the room temperature strength after excursion to high temperatures, which is a desirable property for the intended application of bonding heat-shielding tiles to a GR/PI structural part.

Additional flatwise tensile measurements need to be made at several points in the temperature range 600° - 700°F, both with SIP and without SIP. Also, Owens-Illinois Type 100 and Type 908 glass resins will be investigated to see if these resins can yield even better results.

Material loss occurred at 700°F with the 560 mastic mixture in the flatwise tensile tests. RTV566 glass resin mixtures will also be studied, since RTV566 has an even lower concentration of volatiles than RTV560 and thus may provide additional high temperature strength.

Cryogenic measurements will be made on the materials under investigation, both for lap shear tests and flatwise tensile tests.

Cycling tests to study performance and durability are also planned.

APPENDIX B

EXPERIMENTAL RESULTS

MARCH 1978 THROUGH JUNE 1978

Internal Letter

Rockwell International

Date: . 5 October 1978 No: .

TO: (Name, Organization, Internal Address)
. Kenneth E. Smith
. 041, Dept. 190-900, SL48
.

FROM: (Name, Organization, Internal Address, Phone)
. C. L. Hamermesh
. 083, Dept. 020, A12
.
. 253-2196

Subject: . Status Report - March 1, 1978 to Sept. 30, 1978.
"Adhesive for Bonding Reuseable Insulation (RSI) Tiles to Graphite/Polyimide Structure for Advanced Space Transportation Systems"
Contract NAS1-15152, IDWA M19557-SC 30016-6173

Previous work up to March 1, 1978, showed that the high temperature working range of RTV560 mastic could be increased from its design limit of 500°F up to 700°F by addition of 30% by weight of Type 650 (Owens Illinois Glass Resin) which is a sesquisiloxane type polymer (Fig. B-1). Problems with the lack of reproducibility of Type 650 glass resin mixtures and the short shelf life of the resin alone (3-6 weeks) made it necessary to replace it with Type 908 Glass Resin which has 50% silicon and oxygen, versus 80% silicon and oxygen for Type 650. This was done as the result of conversations with personnel from Owens-Illinois which revealed that GR 650 is not a shelf stable material and in which they recommended a switch to 908 or 100.

Lap shear tests indicated that the optimum concentration of Type 908 glass resin is 25% by weight in the mastic. After mixing this combination consistently produces a smooth mastic with long shelf life, whereas many of the RTV560-GR650 mixtures became lumpy and unspreadable within a few hours. Type 100 glass resin (60% silicon and oxygen) was also studied via lap shear tests. It was more difficult to work with than 908, but less so than 650. Strengths were equal to but no better than 908 mastic mixture.

Mastic Preparation: RTV560-25% GR908

The method of preparation is the same as was developed for the RTV560-GR650 mastic. The glass resin is dissolved in acetone, the acetone solution is mixed into the RTV560, the excess solvent is evaporated at room temperature with frequent stirring, and the mastic mixture is then kept covered until used, and the dibutyl tin dilaurate catalyst is added prior to application. About twice as much catalyst is used as for RTV560 alone (i.e. 40 drops catalyst per 100 g of RTV560 in the mixture). The adhesive after curing is still very rubbery, not brittle (some cured samples with RTV560-GR650 were brittle).

Flatwise Tensile Testing

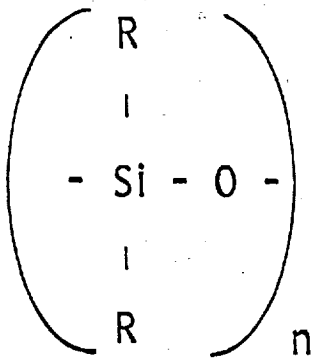
Flatwise tensile tests confirm the capability of the mastic mixture to perform at 700°F and then withstand repeated cycling between -250° and +700°F. At 700°F, the mastic mixture showed a sixfold increase in strength over the RTV560 control specimens in a composite-to-composite bond **(4.0 psi vs. 0.6 psi, average** of 5 tests). In testing the SIP-tile bond at 700 F and at room temperature after excursion to 700°F for 30 minutes, RTV560 failed cohesively in all specimens, while the RTV560-GR908 mixture showed tile failure in all specimens (FWT strength for the tile is 8-20 psi). Cycling tests were performed on sandwiches of GR/PI-Adhesive-Tile-Adhesive-GR/PI. (GR/PI is graphite/polyimide composite). One cycle is: RT →700°F (30 min) →RT (cold block) →-250°F (1 hour) →RT →700°F (30 min.) →RT (cold block). The sandwiches were then bonded to brass cylinders with RTV560 and the flatwise tensile tests were performed at room temperature. Two sets of 3 samples each were run **(Table B-1). The failure mode** for RTV560 was largely cohesive failure at the tile-composite interface while the RTV560 + 25GR908 showed deep tile failure in most cases and little or no cohesive failure.

The most conclusive data showing the capability of the RTV560-GR908 mixture is presented in **Table B-1. The earlier data now discussed** also shows this, but less dramatically. This is because of two problems with FWT tests with these materials: (1) the expansion coeffficient of GR/PI composite is so different from that of the brass cylinders that the brass-composite bond is subjected to severe stress and fails at low psi, (2) the tile FWT strength is variable and very low (8-20 psi). Thus, the failure mode becomes more important than the psi values in analyzing this **data (Tables B-2 and B-3).**

In an attempt to evaluate the composite-composite bond strength in a flatwise tensile test after cycling, sandwiches of composite/adhesive/composite were made and cycled once, then bonded to brass cylinders with RTV adhesive. Most of the samples failed at the composite/brass interface. One RTV560 control sample failed at 13.3 psi in the composite/composite interface. One RTV560 + 25GR908 sample failed at 22.2 psi at the composite/composite interface. Upon examination, the RTV560 in all samples was tacky after cycling, while the glass resin mastic was not. It appeared unaffected by cycling.

C. L. Hamermesh

RTV 560 - SILICONE



GLASS RESIN (OWENS ILLINOIS) - SESQUISILOXANE

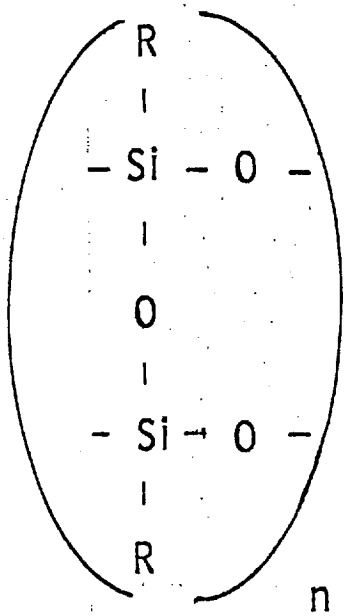


Figure B-1

Table B-1. FWT Cycling Tests (psi)

One Cycle		Two Cycles	
RTV560	RTV560 + 25GR908	RTV560	RTV560 + 25GR908
Set 1			
12.3	16.2	12.6	13.8
14.5	26.1	12.2	15.3
*	17.6	11.9	12.6
Set 2			
5.8	13.3	10.0	17.6
2.1	12.1	9.7	16.9
10.6	15.8	12.6	15.4

*Tile failed during loading

Averages of all results:

No. of Cycles	RTV560	RTV560 + 25GR908
1	9.1	16.9
2	11.5	15.3

Table 3-2. FWT Test, 700°F, 700° Excursion

Sample arrangement: BRASS-ADHESIVE-BRASS-SIP - ADHESIVE-TILE - ADHESIVE-SIP-ADHESIVE-BRASS

RTV560*	Temperature	RTV560 + 25GR908 **
13.8	700°F (30 min)	13.9
12.8	700°F (60 min)	11.0
13.4	R.T. (700° Exc. for 30 min)	10.7
12.1	R.T. (700° Exc. for 60 min)	12.1

* cohesive failure in all specimens

**tile failure in all specimens

Table B-3. FWT Test - 700° Excursion, 30 min., RT Measurement

Sample arrangement: BRASS/ADHESIVE/SIP/ADHESIVE/COMP/ADHESIVE/ COMP/ADHESIVE/SIP/ADHESIVE/BRASS

RTV560, psi	RTV560 + 25GR908, psi
17.6*	20.7**
9.5**	17.9**

700°F (30 min), tested at 700°F:

RTV560	RTV560 + 25GR908
2.7**	3.5**
2.3**	2.5**

* Failure in SIP - Composite Bond

** Failure in Composite-Composite Bond

APPENDIX C

SPECIFICATION OUTLINES

PREPARED BY	CODE IDENT. NO.: 03953	NUMBER MB0120-
R.L. Long	Space Division Rockwell International 12214 Lakewood Boulevard Downey, California 90241	TYPE Material
APPROVALS	**SPECIFICATION**	DATE
		SUPERSEDES SPEC. DATED:
		REV. LTR. / PAGE 1 of

TITLE

LOW VOLATILE CONDENSIBLE MATERIAL, TWO-PART,
-300 TO 700°F ADHESIVE

TABLE OF CONTENTS

1. SCOPE

This specification establishes the requirements for a low volatile condensible material, two-part room temperature curing, -300 to 700°F service adhesive system.

2. APPLICABLE DOCUMENTS

The latest issues of the following documents form a part of this specification to the extent specified herein. In case of a conflict between these documents and this specification, this specification shall prevail.

Federal Specification MMM-A-132	Adhesives, Heat Resistant, Airframe Structure, Metal to Metal
Military Specification MIL-A-9067	Adhesive Bonding, Process and Inspection, Requirements for
NASA Specification SP-R-0022	General Specification, Vacuum Stability Requirements of Polymeric Material for Spacecraft Application
ASTM C 177	Thermal Conductivity of Materials by Means of the Guarded Hot Plate, Test for
ASTM D 2240	Identation Hardness of Rubber and Plastics by Means of a Durometer, Test for

3. REQUIREMENTS

3.1 Form. The adhesive shall be supplied in a two-part system consisting of a compounded base resin with fillers, and a separate curing agent, catalyst. The base resin shall contain no solvents.

3.2 Mixing. The adhesive shall be mixed in accordance with the adhesive manufacturer's instructions.

3.3 Application. The mixed adhesive shall be capable of being applied to metal, composite, or ceramic surfaces at temperatures between 60 and 90°F, and at relative humidities between 30 and 70 percent.

3.4 Pot Life. The pot life, or adhesive working life, shall be at least 1 hour at a temperature of 75 ± 5°F for a 100-gram mixture of adhesive and catalyst.

3.5 Cure. The adhesive mixture shall be capable of meeting the requirements of 3.6, 3.7, and 3.8 after being cured for hours minimum at 75 ± 5°F.

3.6 Mechanical Properties. The cured adhesive shall meet the requirements of Table I.

3.7 Physical Properties. The cured adhesive shall meet the requirements of Table II.

3.8 Volatile Condensible Materials. The cured adhesive mixture shall meet the requirements of Specification SP-R-0022.

3.9 Storage Life. Storage life shall be at least six months for the separate components when stored at ambient temperatures not exceeding 80°F. Storage life is defined as the length of time, starting with the date of manufacture, during which the material will meet all the requirements of this specification.

Table I. Mechanical Properties

Test	Specimen Conditioning	Test Temperature (°F)	Minimum Average Value*	Test Method Paragraph
Lap Shear Strength (psi)	30 ± 5 min at test temperature	-300 ± 10		4.5.2
		75 ± 5		4.5.2
		400 ± 10		4.5.2
		700 ± 10		4.5.2
	Salt spray 30 days at 75 ± 5 °F	75 ±5		4.5.2.1
	Humidity 30 days at 120 ± 5°F and 95 to 100% relative humidity	75 ± 5		4.5.2.2
Peel Strength (lb/in. width)	None	75 ± 5		4.5.3
Hardness Shore A	48 ± 1 hours at 75 ± 5°F	75 ±5		4.5.5

*All values are minimum for the average of four specimens with no individual value less than 90% of the value listed.

Table II. Physical Properties

Test	Test Temperature (°F)	Minimum Average Value	Test Method Paragraph
Thermal conductivity (Btu/hr-ft^2-°F/in. minimum)	-300 to 700		4.5.4

4. QUALITY ASSURANCE

4.1 Qualification.

4.1.1 Material submitted for qualification to this specification shall meet all requirements of this specification.

4.1.2 If there is any change in the formulation of the material originally qualified to this specification, a new manufacturer's designation shall be assigned and the material shall be resubmitted for qualification.

4.2 Acceptance.

4.2.1 For purposes of sampling, inspection, and tests, a "lot" shall consist of all material supplied to one Purchase Order and submitted for acceptance at one time; a "batch" shall be that quantity of material compounded and manufactured at one time. Each batch in each lot shall be tested for conformance to the acceptance requirements of 4.2.2.

4.2.2 Acceptance shall be defined as the minimum number of tests that shall be performed on each batch within a shipment. The tests performed shall be as follows:

- Lap shear strength 75 $\pm$ 5°F
- Lap shear strength 700 $\pm$ 10°F
- Peel strength at 75 $\pm$ 10°F

4.3 Certification. A certified report from the supplier shall accompany each shipment stating conformance to the acceptance requirements of 4.2.2. This report shall include actual test data for the acceptance tests required in 4.2.2 and shall also include this specification number, Purchase Order number, batch number or lot number, manufacturer's designation, and date of manufacture.

4.4 Responsibility for Inspection and Testing. The supplier is responsible for the performance of all inspection and testing specified herein. The supplier may use his own facilities or those of a commercial laboratory. Rockwell International reserves the right to perform any of the inspection and testing set forth in this specification, where such are deemed necessary to assure compliance with specification requirements.

4.5 Test Methods.

4.5.1 Surface Preparation. Both stainless steel and aluminum specimens shall have faying surfaces to be bonded prepared per the applicable method in MIL-A-9067.

4.5.2 Lap Shear Strength. Aluminum and stainless steel specimens and test methods shall conform to MMM-A-132. The aluminum alloy shall be base 2024-T3 or -T81 and shall be used for the 75 $\pm$ 5°F and colder tests. The stainless steel shall be and shall be used for the 400°F and higher temperature tests. All tests conducted at other than room temperature shall be soaked at the test temperature for a 35 $\pm$ 5 minutes before the initiation of test.

4.5.2.1 Salt Spray. Salt spray exposure panels shall be fabricated and tested at 75 ± 5°F in accordance with MMM-A-132 using bare aluminum alloy 2024-T3 or -T81.

4.5.2.2 Humidity. Humidity exposure panels shall be fabricated and tested at 75 ± 5°F in accordance with MMM-A-132 using bare aluminum alloy 2024-T3 or -T81.

4.5.3 Peel Strength. Peel specimens shall be 0.025-inch-thick aluminum (1 inch wide by 12 inches long) bonded to 0.063-inch-thick aluminum (1 inch wide by 10 inches long). The aluminum alloy shall be bare 2024-T3 or -T81. Style 112 glass cloth may be used as a scrim cloth to maintain a uniform bondline thickness. The cured specimen shall be installed in the fixture shown in Figure 1, or equivalent, and tested as shown in Figure 2. The rate of Read travel on the testing machine shall be 5 to 6 inches per minute. All tests shall be conducted at 75 ± 5°F.

4.5.4 Thermal Conductivity. The thermal conductivity shall be determined per ASTM-C-177.

4.5.5 Hardness. A Shore A hardness instrument shall be used. Three readings shall be taken at 75 ± 5°F after 48 hours from application per ASTM D 2240.

4.5.6 Volatile Condensible Materials. The volatile condensible materials shall be determined per NASA specification SP-R-0022.

5. PREPARATION FOR DELIVERY

5.1 Packaging. The material shall be packaged in suitable containers for shipping in accordance with standard commercial practice. The shipping containers shall be so constructed as to assure safe transportation to the point of delivery.

5.2 Marking. Each shipping container shall be permanently marked with the following information:

MATERIAL: Low Volatile Condensible Material, Two-Part Silicone Adhesive

ROCKWELL INTERNATIONAL SPECIFICATION NO.: MB0120-

MANUFACTURER'S NAME: _____

PRODUCT IDENTIFICATION: (Resin, Catalyst, Primer, etc.)

LOT NO. _____ BATCH NO. _____

DATE OF MANUFACTURE: _____

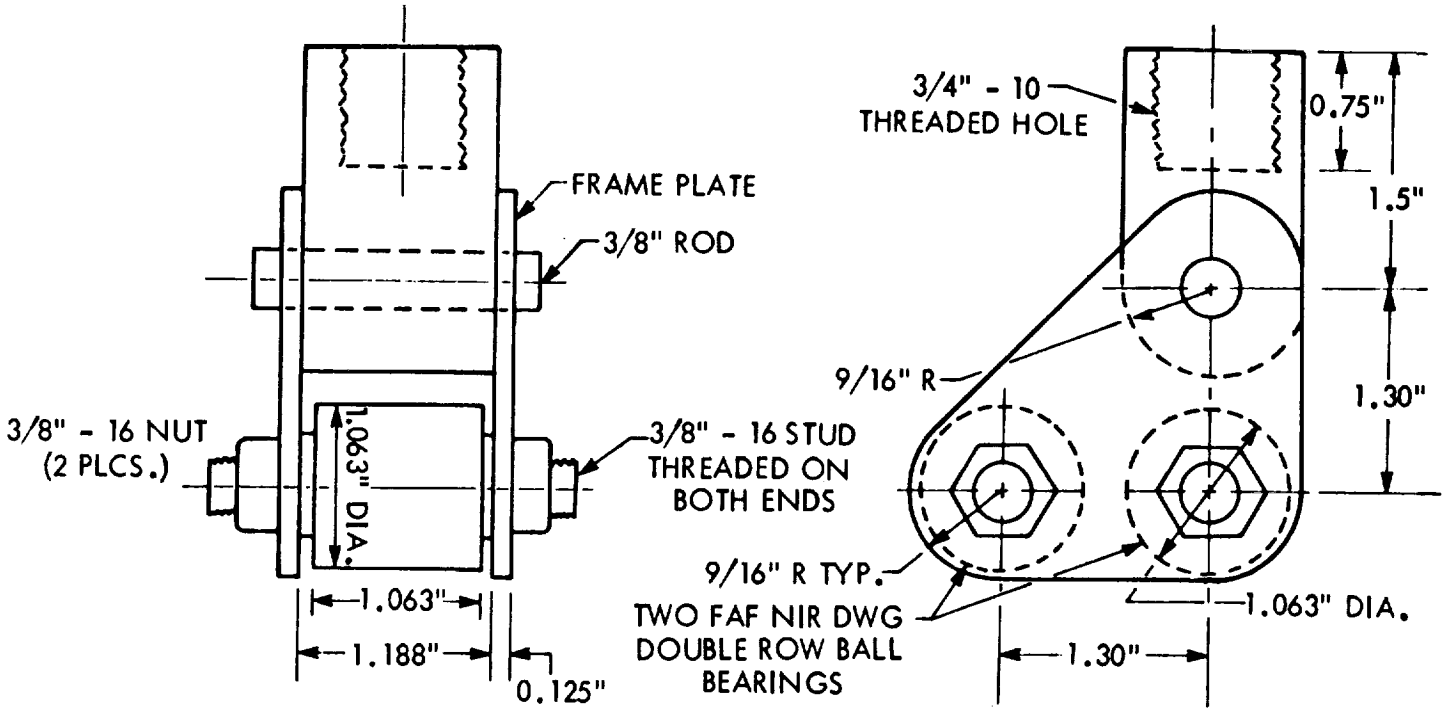


Figure 1. Metal-to-Metal Peel Test Fixture

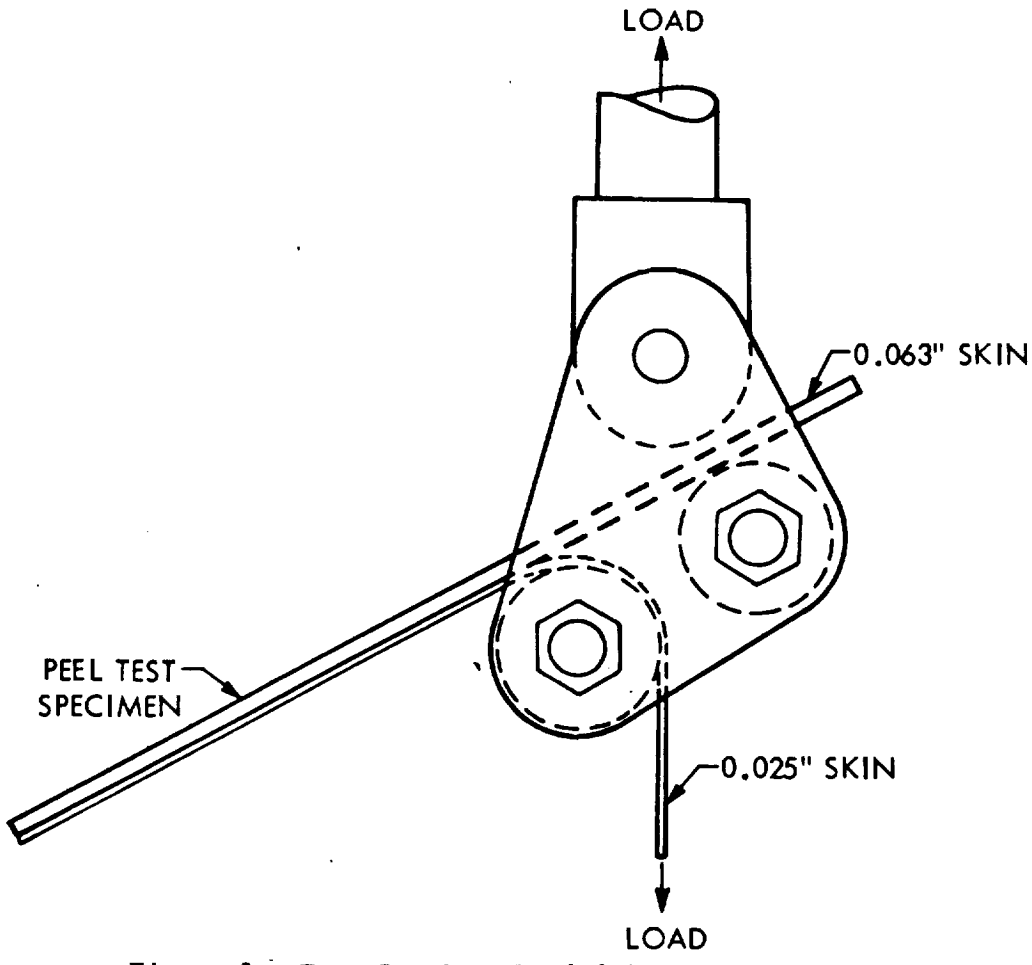


Figure 2. Test Specimen Loaded in Peel Test Fixture

PREPARED BY	CODE IDENT. NO.: 03953	NUMBER MA0106-
R. L. Long	Space Division Rockwell International 12214 Lakewood Boulevard Downey, California 90241	TYPE Process
APPROVALS	**SPECIFICATION**	DATE
		SUPERSEDES SPEC. DATED:
		REV. LTR. / PAGE 1 of 14

TITLE

ADHESIVE BONDING SILICA REUSABLE
SURFACE INSULATION (RSI) SYSTEM

DRAFT COPY

TABLE OF CONTENTS

1. SCOPE

1.1 General. This material processing specification (MPS) establishes the engineering requirements for an adhesive bonded silica thermal protection system (TPS) using a room temperature curing adhesive.

1.2 Applicability. The MPS is applicable, but not restricted to bonding silica reusable surface insulation (RSI) tile, and subassemblies ~~and filler-bar assemblies~~ to Shuttle orbiter interface mold line (IML) surfaces as defined in ML0301-0010.

2. APPLICABLE DOCUMENTS AND MATERIALS

2.1 Documents. The latest issues of the following documents form a part of this MPS to the extent specified herein. In case of conflict between referenced documents and this MPS, the requirements of this MPS shall prevail.

FED-STD-601	Rubber, Sampling and Testing
MMM-A-132	Adhesive, Heat Resistant, Airframe Structural, Metal to Metal
ASTM D 297	Tension Test of Flat Sandwich Construction in Flatwise Plane
MF0004-045	Reusable Surface Insulation (RSI) - Tile Surface Features
MF0004-048	Close-out Tile and Composite Tile, Surface Features

2.2 Materials.

TT-M-261	Methyl Ethyl Ketone
ST0210GB0002	Trichloroethane, 1,1,1 Stabilized, Vapor Degreasing and Solvent Flusing Grade
MB0125-050	Primer, Silicone, for Low Volatile Content Applications
MB0120-XXX	Low Volatile Condensible Material, Two-Part, -300 to 700°F Adhesive

3. REQUIREMENTS

3.1 Materials.

3.1.1 Silica Tile and Bonding Materials. The silica tile and the bonding materials shall be as specified by the engineering drawing for reusable surface insulation. See Figure 1 for the configuration of a typical thermal protection system installation.

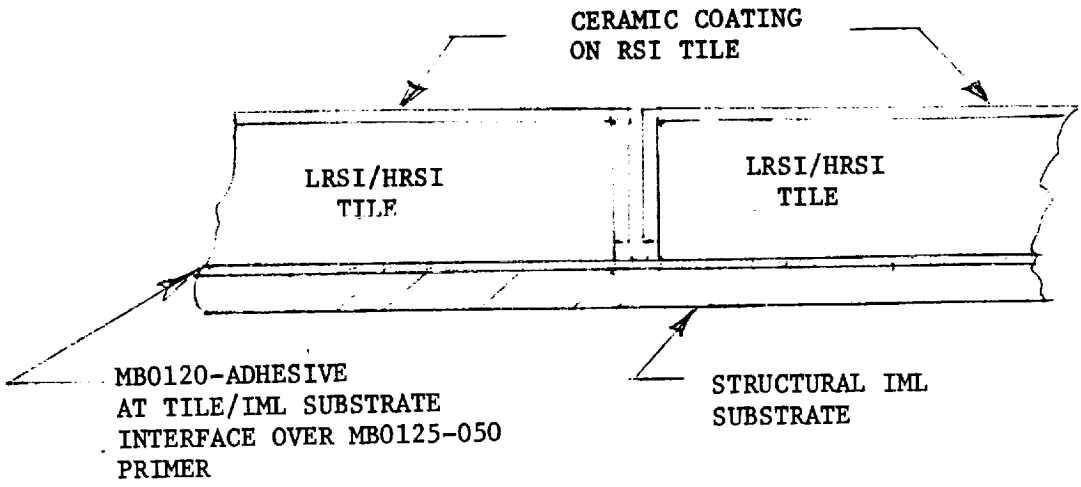


Figure 1. Typical LRSI/HRSI Tile Installation

3.1.2 Adhesive. The adhesive system shall consist of a room temperature curing material conforming to MB0120-XXX.

3.2 IML Surface Fairing, Structural Gap Sealing and Fairing Non-Conforming Steps Between Panels of the IML Structure.

3.2.1 IML Surface Fairing. Waviness in the interface mold line or structural substrate surface shall not exceed the requirements defined on the engineering drawing (i.e., substrate smoothness versus waviness criteria).

3.2.1.1 If required, low areas/localized depressions in the acreage IML that exceed the surface waviness requirement shall be faired (filled) with MB0120-XXX adhesive to conform to the applicable IML smoothness requirement.

3.2.1.1.1 Prepare affected zones by: (1) scuff-sanding nonmetallic surfaces per 3.4.1.1 and (2) solvent wiping surfaces per 3.4.1.3.

3.2.1.1.2 Apply MB0125-050 adhesive primer per 3.4.2, 3.4.3, and 3.4.4.

3.2.1.1.3 Apply and fair the filling compound into low areas by screeding to appropriate thickness using MB0120-XXX adhesive per 3.5.1 followed by curing per 3.6.2.

3.2.2 Gap Sealing. Butt-joint gaps in the IML structure shall be sealed with MB0120-XXX adhesive to insure that vacuum-bag bonding of the RSI tile subassemblies or arrays can be achieved.

3.2.2.1 Foreign matter shall be removed from gaps at joints in the IML structure using nonmetallic scrapers.

3.2.2.2 Gaps in nonmetallic areas shall be: (1) scuff-sanded per 3.4.1.1 and (2) solvent-wiped per 3.4.1.2.

3.2.2.3 Gaps shall be primed with MB0125-050 adhesive primer per 3.4.2, 3.4.3, and 3.4.4.

3.2.2.4 Gaps shall be sealed using MB0120-XXX adhesive per 3.5.1, followed by curing per 3.6.2.

3.2.3 Step Fairing. When required, non-conforming steps at butt-joints and doublers in structural IML panels shall be faired to provide conformance to the applicable IML smoothness requirements defined on the engineering drawing.

3.2.3.1 Non-conforming butt-joints, steps (and adjacent areas) in the IML nonmetallic structure shall be: (1) scuff-sanded per 3.4.1.1 and, (2) solvent-wiped per 3.4.1.2.

3.2.3.2 Areas to be faired shall be primed with MB0125-050 adhesive primer per 3.4.2, 3.4.3, and 3.4.4.

3.2.3.3 Steps shall be faired using MB0120-XXX adhesive per 3.5.1 followed by curing per 3.6.2. Fairing runout to the IML shall be in the approximate proportion of 100 mils for each mil of rise.

3.3 Tile Mismatch.

3.3.1 Tile IML Defect Filling. Visible defects in tile IML surfaces that conform to the allowable defect depth/width/length criterion defined in MF0004-045 or MF0004-048 shall be treated as follows:

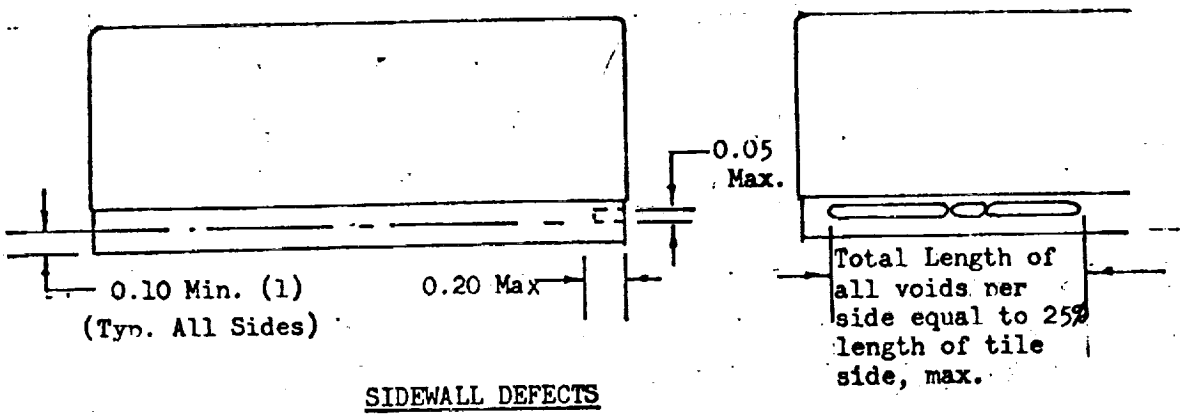
3.3.1.1 Vacuum clean the entire IML surface of affected tile.

3.3.1.2 Visible tile IML defects (or IML surface voids) that are determined to be totally within the bond interface shall be filled with MB0120-XXX and faired flush with the tile IML surface while wet. The filling of the defects, shall be accomplished concurrently while applying the wet tie-coat using the same mix of adhesive (Case I, Figure 2).

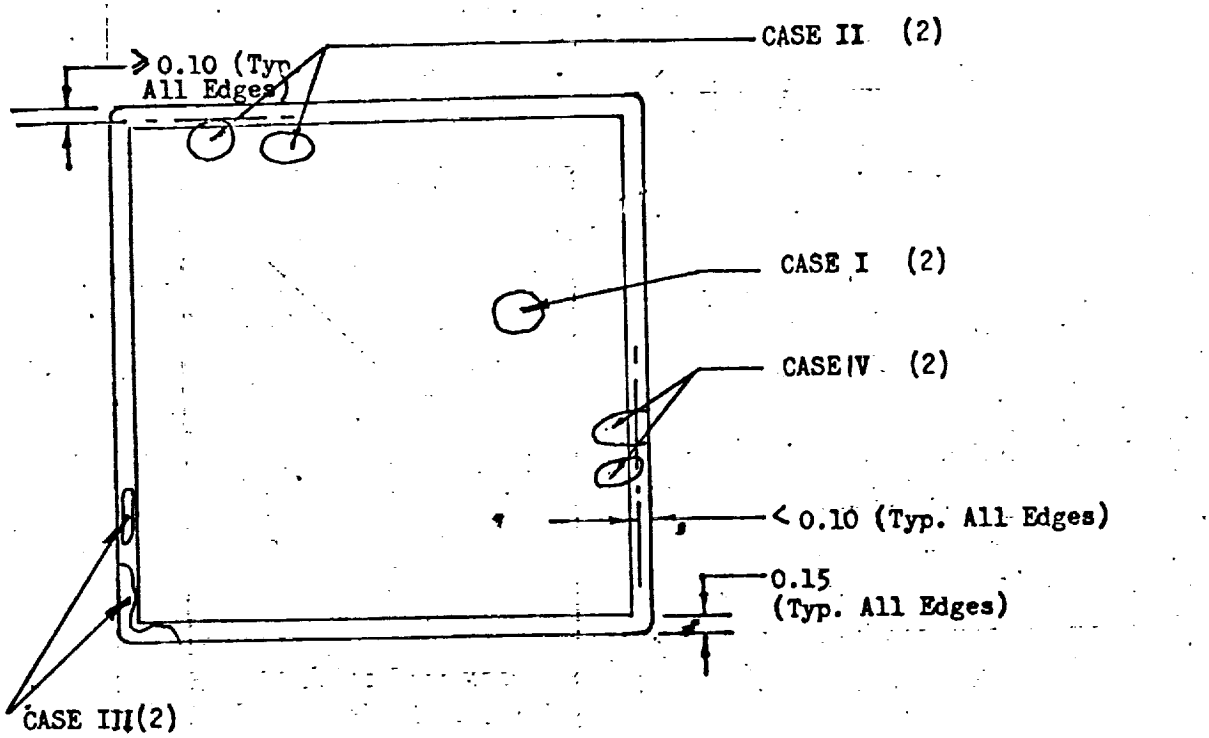
3.3.1.3 Visible tile IML defects (or IML surface voids) determined to be totally contained within the 0.56 to 0.1 zone shall be processed per 3.3.1.2 (Case II, Figure 2).

3.3.1.4 Visible tile IML defects (or IML surface voids) totally contained within the 0.15 to edge zone shall not be filled if $\leq$ 0.15 inch in depth (Case III, Figure 2).

IML, SIDEWALL, AND EDGE DEFECTS - TILES



SIDEWALL DEFECTS



IML/ EDGE DEFECTS

(1) Sidewall voids which extend below 0.10 minimum dimension shall be evaluated as an edge defect.

(2) Not to exceed 25 percent of tile thickness.

Figure 2. Adhesive Filling Requirements

3.3.1.5 Visible tile IML defects (or IML surface voids) that cross the seal zone shall be filled with MB0120-XXX and faired flush with the tile IML surface while wet. Cure until a firm tack-free surface is attained prior to further processing/handling of tilt. The fill shall be flush to within 0.010 inch below the tile IML surface when cured. If flushness to within 0.010 inch below the tile IML surface has not been achieved, refill the zone over the first fill using MB0120-XXX and fair flush to the tile IML surface while wet. Refilling the defects shall be accomplished concurrently while applying the wet tie-coat using the same mix of adhesive (Case IV, Figure 2).

3.3.1.6 Sidewall defects (or sidewall voids) that are contained in a zone below the coating terminator, but above 0.1 inch from the tile IML and measure (1) 0.05 inch maximum width by 0.2 inch maximum depth by 25-percent maximum length of the tile side in a tile of 0.6 inch in thickness and (2) 0.15 inch maximum width by 0.2 inch maximum depth by 25-percent maximum length of the tile side in a tile 0.6 inch in thickness, shall not be filled (Figure 2).

3.3.2 Tile Mismatch with Corresponding IML Surface. Mismatch of tile to the structural IML substrate shall not exceed the requirements shown on the engineering drawing. If tile/IML surface mismatch is predicted, such areas shall be adjusted by any combination of the following methods:

3.3.2.1 Machining the uncoated (backface) surfaces of the tile per the engineering drawing prior to application of tile to substrate.

3.3.2.2 Using a pre-cast, cured film (or shim) of adhesive which is adhered to the IML surface in a secondary bond application per 3.3.3.1.1 through 3.3.3.1.3.

3.3.3 Tile/OML Steps. OML steps shall not exceed the requirements shown on the engineering drawing.

3.3.3.1 If out-of-tolerance tile/OML step mismatch is predicted, such areas shall be adjusted by any combination of the following methods:

3.3.3.1.1 Machining the uncoated (backface) surface(s) of the tile per the engineering drawing prior to application of adhesive to the tile.

3.3.3.1.2 Screeding localized depressions per 3.2.1.1.1 through 3.2.1.1.3.

3.3.3.1.3 Using a pre-cast, cured film (or shim) of adhesive which is adhered to the IML surface in a secondary bond application per 3.3.3.1.1 through 3.3.3.1.2.

3.3.3.2 The pre-cast films shall be fabricated to the appropriate thicknesses using MB0120-XXX adhesive per 3.5.1, followed by curing per 3.6.2.

3.3.3.3 The IML surface zones to which the pre-cast adhesive shims are to be applied shall be: (1) scuff-sanded per 3.4.1.1 if a structural nonmetallic surface, and (2) shall be solvent-wiped per 3.4.1.3.

3.3.3.4 The affected IML surface zones shall be primed with MB0125-050 adhesive primer per 3.4.2, 3.4.3, and 3.4.4.

3.3.3.5 The pre-cast shims shall be bonded as required to applicable IML surface zones with MB0120-XXX adhesive per 3.5.1.1. The bond of the shim to the IML surface shall consist of one thin continuous wet coat of MB0120-XXX adhesive cured per 3.6.2.

NOTE: Thickness measurements for the thin continuous wet-film secondary bond are not required.

3.4 Priming Substrates with MB0125-050 Adhesive Primer.

3.4.1 Surface Preparation. The types of surfaces defined below shall be prepared for subsequent TPS bonding as follows:

3.4.1.1 MB0120-XXX Screeded Surfaces.

3.4.1.1.1 Dry sand to remove "gloss" and/or roughen the adhesive surface and fair edges (if necessary) using 240 grit (or finer) abrasive paper.

3.4.1.1.2 Dry wipe using clean cheesecloth (or equivalent).

NOTE: If the sanded surface subsequently becomes soiled or contaminated prior to bonding, solvent wipe appropriate zones using TT-M-261 methyl-ethyl-ketone, wiping to dryness using clean cheesecloth (or equivalent), followed by air drying for 16 hours (minimum).

3.4.1.2 MB0120-XXX Bonded Heat Sink Pad Surfaces.

3.4.1.2.1 Process per 3.4.1.1 if required to remove "gloss" from cured adhesive bond interface.

3.4.1.3 Non Koropon-Primed Polyimide and Polyimide Graphite Structure Surface.

3.4.1.3.1 Scuff-sand (dry) to remove "gloss" using 180 grit (or finer) abrasive paper.

3.4.1.3.2 Solvent wip using ST02.0GB0002,1,1,1 trichloroethane followed by TT-M-261 methyl-ethyl-ketone; wipe to dryness using clean cheesecloth.

3.4.1.3.3 Air dry one hour (minimum) to four hours (maximum) prior to applying silicone primer.

3.4.2 Applications.

3.4.2.1 All nonmetallic structural substrate surfaces shall be primed with MB0125-050 adhesive primer prior to the subsequent application of MB0120-XXX adhesive for the following:

a. IML surface fairing

b. IML gap sealing/fairing

c. IML structural step fairing

d. Bonding shims for adjustments in tile OML steps

e. Bonding tiles to the IML substrate.

NOTES: 1. RSI tile shall not be primed with MB0125-050 adhesive primer.

2. Adhesive coating, sealants, filling fairing compounds already present on substrate bond surfaces shall not be primed with MB0125-050 adhesive primer.

EXCEPTIONS: a. Primer applied to adhesive used as a sealant in butt-joint gaps $\leq$ 0.250 inch is permitted.

b. A nominal 0.125-inch overlap of primer onto previously applied adhesive is permitted.

3.4.3 Cure of Applied Primer.

3.4.3.1 The MB0125-050 adhesive primer shall be allowed to cure (hydrolyze) for two hours minimum at at ambient temperature/humidity conditions prior to the subsequent application of the MB0120-XXX adhesive.

3.4.3.2 Primer left to cure in excess of 24 hours shall be removed per 3.4.1.2 and the applicable substrate zone reprimed per 3.4.2.1.

3.4.4 Appearance.

3.4.4.1 The adhesive primer applied to structural IML substrate surfaces shall exhibit evidence of having been applied in a continuous film.

3.5 Application of MB0120 Adhesive.

3.5.1 Fairing, Sealing, Shimming Applications and RSI Tile Bonding.

3.5.1.1 MB0120-XXX adhesive shall be used for the applications defined in a through e. The adhesive shall exhibit a continuous film, wetting all surfaces to which it is applied. Such areas shall include, but not be limited to:

a. Screeded IML surface fairing and/or heat sink zones.

b. IML gap sealing zones

c. IML step fairing zones

d. Bonding heat sink pads or shims for adjustments in steps of tile OML.

e. Bonding tile to the IML substrate.

3.5.1.2 The bondlines of TPS composite (i.e., substrate, tile) shall be comprised of:

3.5.1.2.1 Tile to IML Bonds. A one-coat application of MB0120-XXX adhesive applied to the IML substrate when bonding the tile to the substrate. The substrate bondline shall have a continuous wet-film thickness of MB0120-XXX adhesive measuring 5.5 to 9.5 mils thick (0.075 $\pm$ 0.002 inch).

4. QUALITY ASSURANCE

4.1 Personnel Qualification. All bonding operations shall be performed by qualified personnel.

4.2 Process Qualification.

4.2.1 The bonding procedure for the process shall be qualified prior to acceptance of production binding for each combination of substrate and adhesive used. Qualification shall consist of documented evidence that the process is capable of producing bonds that will meet all requirements of this MPS. Any change in procedure, substrate, or adhesive will require requalification.

4.2.2 Lap shear test specimens shall be fabricated under the same conditions and at the same time as the bonding of a tile (RSI) array and shall meet the requirements of Table I. The primers and adhesive used to fabricate lap shear specimens shall be the same as those used to install RSI.

4.2.2.1 A minimum of eight lap shear specimens shall be tested for each temperature to qualify the process for the strengths specified in Table I.

4.2.3 A minimum of five Shore A hardness readings shall be taken at room temperature to qualify the procedure for the hardness requirements of 3.6.2.

4.2.4 Flatwise tensile composite specimens shall be fabricated as shown in Figure 4 and shall meet the requirements of 3.6.3.

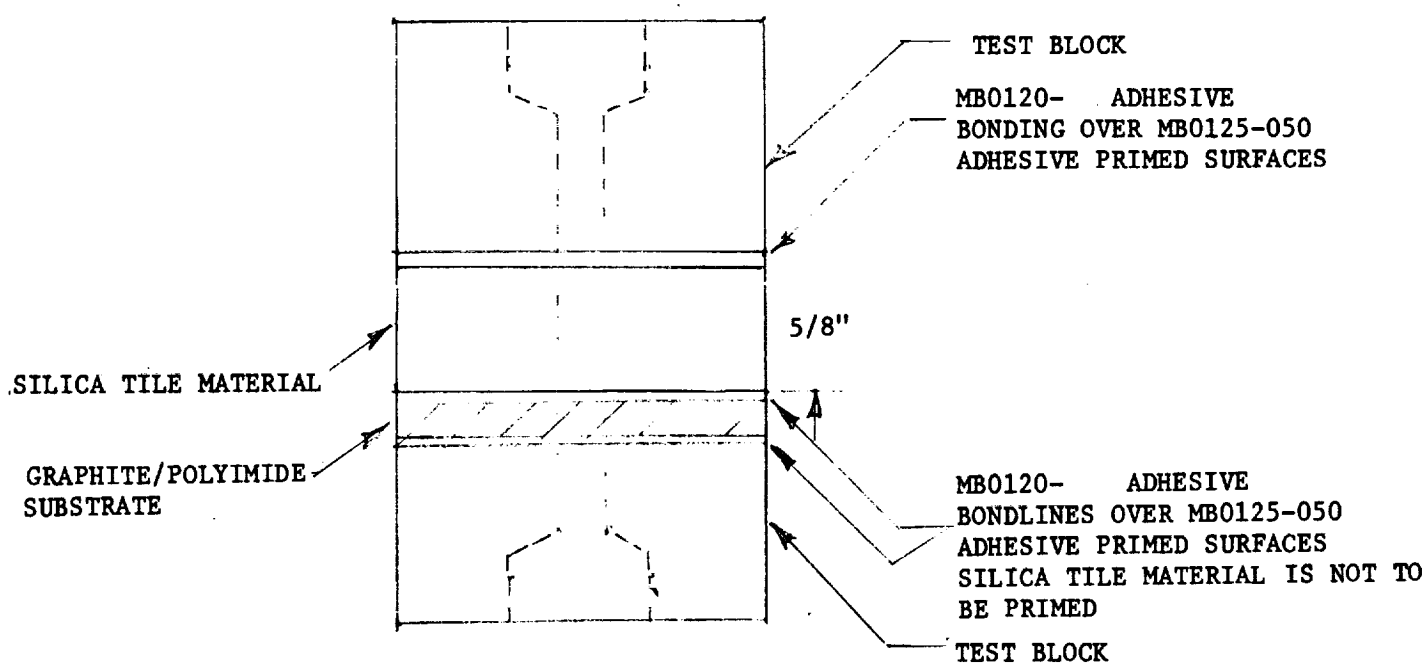


Figure 4. Cylindrical Flatwise Tensile Test Composite

3.5.1.3 In each bondline, the total amount of adhesive shall be applied to the substrate when bonding the tile to the IML.

3.5.1.4 The temperature/humidity limitations as related to work life for all adhesive applications shall be governed by Figure 3 requirements.

3.6 Adhesive Used for Fairing, Sealing, Shimming Applications and For Bonding Tile to the IML Substrate. When cured the adhesive used for the fairing, sealing, shimming applications and for bonding tile to the IML substrate shall meet the following requirements:

3.6.1 Cured Adhesive Strength (for Process Qualification Only). The cured adhesive shall meet the strength requirements of Table I by evaluating adhesive primed steel lap shear specimens that have been prepared, bonded, and cured concurrently with the qualification test part and tested per 4.2.2.

Table I. Lap Shear Strength

Test Temperature (°F ± 10)	Minimum Average Value (psi)
75	
700	

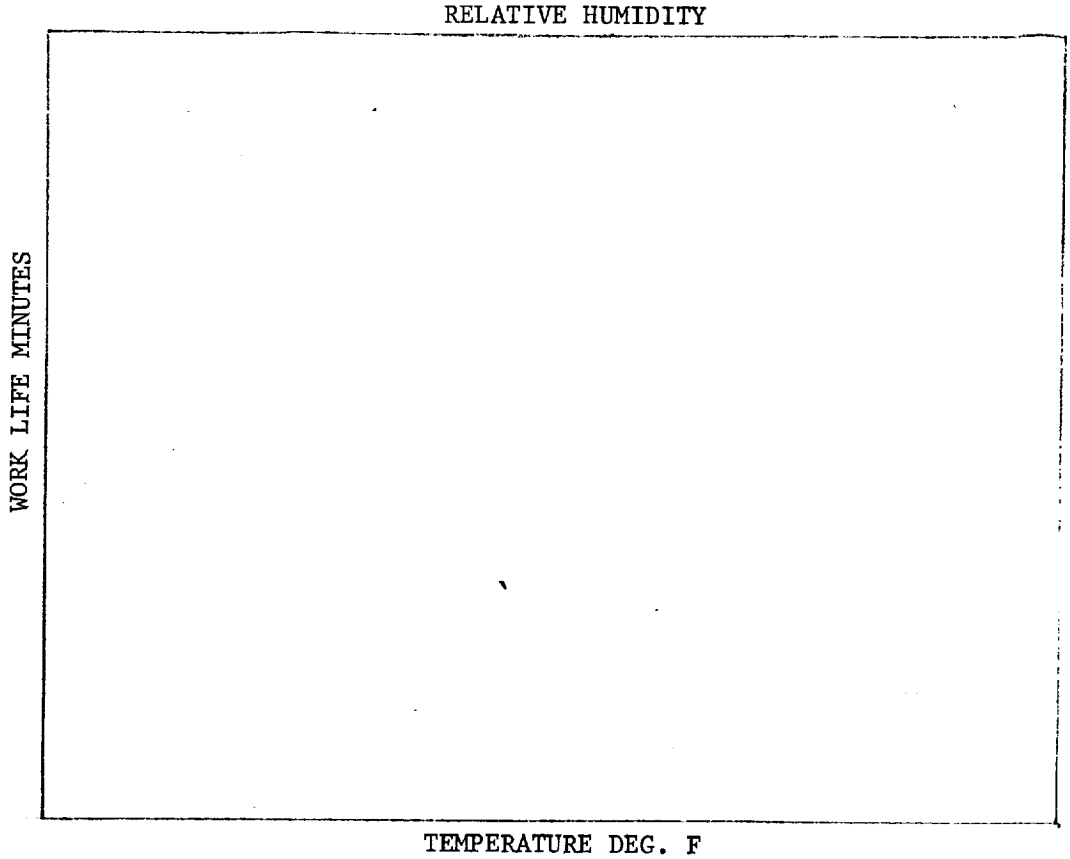


Figure 3. MB0120- Work Life

NOTES:

1. One inch overlap for lap-shear specimens.
2. No individual specimen value shall be less than 90 percent of the average value specified.
3. Hold (soak) at 700°F for 30 minutes minimum prior to testing.
4. Bondline thickness shall be maintained at 0.015 ± 0.005 inch.
5. A minimum of eight lap shear specimens shall be tested at each temperature.

3.6.2 Hardness. The cured adhesive on a test specimen prepared and cured concurrently with the assembly shall have a Shore A hardness of minimum for MB0120-XXX when tested per 4.2.3 and 4.3.1.

3.6.3 Cured Assembly Strength (For Process Qualification Only). The bond strength between the tile and the substrate shall be greater than the flatwise tensile strength of the silicon tile when tested per 4.2.4 at room temperature and 700°F.

3.6.4 Assembly Appearance. The coating on silica tiles shall be free from cracks and chipped areas when examined per 4.4.1.

3.7 Tile Gaps/Steps. Expansion gaps and steps between silica tiles shall be as specified by the engineering drawing.

3.8 Process Quality Validation (PQV) Test. The bonded tile shall not fail when subjected to the PQV tension loading specified (based on tile footprint area) during testing per 4.3.3. The tensile load shall be applied through the centroid of the footprint, except as noted below.

3.8.1 Low Temperature Reusable Surface Insulation (LRSI). The load applied shall be 1.0 ± 0.1 psi. No individual measurement shall exceed 20 mils corrected "true" value. In addition, the maximum difference between the highest and lowest uncorrected peak deflection values at the tile corners (i.e., tilb) shall not exceed 25 mils.

NOTE: When tile configurations do not permit tension testing to the load specified, a qualitative test may be substituted. The load need not be directed through the centroid in these instances.

3.8.2 Mini-Tile. Mini-tiles shall be PQV tested to verify the integrity of the tile to structure loads.

3.8.3 High-Temperature Reusable Surface Insulation (HRSI). The load shall be 2.0 ± 0.1 psi. No individual deflection measurement shall exceed 35 mils corrected "true" value. In addition, the maximum difference between the highest and lowest uncorrected peak deflection values at tile corners (i.e., tilt) shall not exceed 35 mils.

NOTE: When tile configurations do not permit tension testing to the load specified, a qualitative test may be substituted. The load need not be directed through the centroid in these instances.

4.2.4.1 Four specimens each shall be tested at room temperature, 600°F, and 700°F.

4.2.5 PQV testing of bonded tile by the vacuum method shall meet the requirements of 3.8. Frequency, quantity and location shall be determined by Quality Assurance.

4.3 Product Acceptance.

4.3.1 Product acceptance shall be based on evidence of compliance with the requirements of this MPS and the detailed operating and inspection procedures that were used in qualification of the process and by Shore A hardness test specimens that accompany the product. The hardness specimens shall be prepared concurrently with the assembly using the same mixes of adhesive.

4.3.2 A minimum of five Shore A hardness readings shall be made at room temperature to verify process control for product acceptance. Shore A hardness shall meet the applicable requirement of 3.6.2.

4.3.3 PQV testing by the vacuum method shall meet the requirement of 3.8. Method, frequency, quantity, and location shall be determined by Quality Assurance.

4.4 Inspection Methods.

4.4.1 Visual. Visual inspection with the unaided eye (without magnification) shall be used to determine conformance to the visual requirements of this MPS.

4.4.1.1 Substrate. The cleaned substrate surface shall be inspected for conformance to 3.4.1.

4.4.1.2 Primed Surfaces. Primed surfaces shall be inspected for conformance to 3.4.2, 3.4.3 and 3.4.4.

4.4.1.3 Applied Adhesive. Applied adhesive shall be inspected for conformance to 3.5.1.

4.4.1.4 Cured Adhesive. The cured adhesive shall be inspected for conformance to 3.6.2.

4.4.1.5 Bonded Assemblies. The bonded insulation shall be inspected for conformance to 3.6.4.

4.4.2 Lap Shear Testing. The lap shear strength shall be determined per MMM-A-132.

4.4.3 Flatwise Tensile Strength. The tensile strength shall be determined in accordance with ASTM D 297 using cylindrical flatwise tensile specimens (Figure 4).

4.4.4 Hardness. The Shore A hardness shall be measured per FED-STD-601, Method 3021.

5. PREPARATION FOR DELIVERY. Not applicable.

6. NOTES.

6.1 RSI. The RSI is comprised of HRSI and LRSI tile. Each type of tile consists of a low density fibrous silica block coated on all sides except the bond interface with a waterproof ceramic coating. The uncoated (backface) side of the tile is bonded to the substrate as specified by the engineering drawing and Figure 1.

6.2 HRSI Tile. High temperature reusable surface insulation.

6.3 LRSI Tile. Low temperature reusable surface insulation.

6.4 Mini-Tile. LRSI tile diced into smaller segments.

REFERENCES

1. Smith, R. E., Development of an Alternate Orbiter TPS Tile Attachment Subsystem. Rockwell International, Space Division, SD 76-SA-0069 (Feb 1977).

2. Statement of Work "Adhesives for Bonding Reusable Surface Insulation (RSI) tiles to a graphite/polyimide structure for Advanced Space Transportation Systems," Contract NAS1-15152, NASA, Langley Research Center (March 9, 1977).